

**U.S. Fish and Wildlife Service
Region 3
Contaminants Program**

**1992 Sediment Quality Report
for Mark Twain National Wildlife Refuge
Illinois, Iowa and Missouri**



**U.S. Fish and Wildlife Service
4469 - 48th Avenue Court
Rock Island, Illinois 61201
1995**



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for Mark Twain National Wildlife Refuge
Illinois, Iowa and Missouri**

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FINAL REPORT - June 17, 1995

**prepared for
Mark Twain National Wildlife Refuge
Under USFWS Project Number 3N03**

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Abstract

Sediment quality was assessed in 1992 by the U.S. Fish and Wildlife Service at six backwater sites along the Upper Mississippi River. Five of the backwater sites were in Mark Twain National Wildlife Refuge. The Sediment Quality Triad approach was attempted to evaluate heavy metal and nutrient contamination. The Sediment Quality Triad methods included analytical chemistry, benthos assessment and toxicity testing using the Microtox[®] system. Heavy metal concentrations were within or slightly elevated above background limits. Nutrient concentrations were slightly to very elevated. The reference site for the study area was at Skunk Slough in Pool 19. Skunk Slough had abundant infaunal macroinvertebrates with above average heavy metal concentrations and low ammonia concentrations. Keithsburg Division in Pool 18 was identified as a backwater with contaminant problems. Keithsburg Division had a poor assemblage of infaunal macroinvertebrates with above average heavy metal concentrations and the highest concentrations of ammonia for the study area. Ammonia toxicity was the suspected cause of some mortality in sediment elutriate toxicity tests completed at Keithsburg Division. The elutriate toxicity tests were intended to validate the Microtox[®] tests. The Microtox[®] test results were not used because they did not relate to the benthos diversity and chemistry data. GIS analysis indicated that the contaminant problems at the Keithsburg Division backwater were not related to point source pollution such as wastewater discharges into adjacent rivers. The results from this project were used to initiate a multi-year study on non-point source pollution at Keithsburg Division.

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Introduction

This document reports and interprets sediment quality information for selected Upper Mississippi River backwater areas in Mark Twain National Wildlife Refuge (MTNWR) (Figure 1). The sediment quality information includes analytical chemistry, benthic macroinvertebrate diversity and toxicity test results. Point source and non-point source pollution data were cataloged using Geographical Information System (GIS) methods to help identify potential pathways for contaminants. The project was conducted by biologists from the U.S. Fish and Wildlife Service's Rock Island Field Office (Rock Island, IL) as part of the contaminants program for the Service's Division of Refuges and Wildlife.

Background

In 1989, staff from the Rock Island Field Office surveyed sediment chemistry along the Illinois River and Mississippi River in Illinois, Iowa and Missouri (Young 1991). The 1989 data show that organic pollutants such as polychlorinated biphenyls (PCB's), petroleum products, chlordane and similar pesticide compounds were not detected at MTNWR units along the UMR except for Keithsburg Division (Young 1991). Several hydrocarbon compounds (polycyclic aromatic hydrocarbons - PAHs) were detected at low concentrations at Keithsburg Division (Young 1991). The PAHs at Keithsburg Division are believed to be those produced in natural systems by aquatic plants.

The results of the 1989 study indicated some sites in MTNWR had poor sediment quality conditions based on the mortality of test organisms used in toxicity tests (Young 1991). In 1992, several sites in MTNWR with suspected poor sediment quality were re-evaluated in greater detail to more clearly identify the ecological impacts from contaminants.

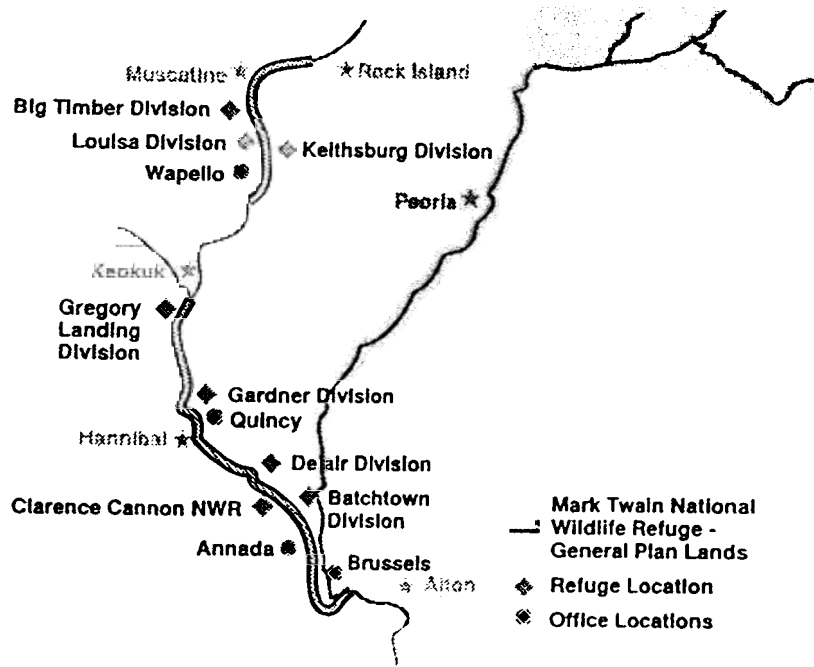


Figure 1. Location of Mark Twain National Wildlife Refuge, Illinois, Iowa and Missouri.

Objectives

The objectives for the re-evaluation of sediment quality are listed below.

1. Measure heavy metal and ammonia concentrations in surficial sediments in selected backwater areas.
2. Examine the substrate for infaunal (substrate dwelling) macroinvertebrate species that are indicators of good sediment quality.
3. Test the toxicity of the sediments with the Microtox[®] system and validate the Microtox[®] tests with elutriate tests.
4. Initiate a GIS database with sediment quality and related environmental information.

Study Sites

The refuge sites re-evaluated in 1992 were Batchtown Division (Pool 25), Clarence Cannon Refuge (Pool 25), Gardner (Pool 21), Keithsburg (Pool 18) and Big Timber (Pool 17) Divisions. A non-refuge study site was established in Skunk Slough (Pool 19) to serve as a reference sampling area. The locations of the study sites are outlined in Figure 2 and related geographic information is in Table 1. Study site maps with the positions of the sampling locations are illustrated on site maps in Appendix A.

Skunk Slough was used as a reference sampling area because it supported diverse populations of benthic macroinvertebrates including pollution sensitive species such as burrowing mayflies (*Hexagenia* species) and fingernail clams (family Spaeriidae). The presence of abundant pollution sensitive organisms served as an indication of good sediment quality for this study.

All of the study sites except Clarence Cannon Refuge are forested backwater complexes with old sloughs, lakes and wetlands. Clarence Cannon Refuge is a network of wetlands within a green tree reservoir.

The study sites are directly adjacent to the Upper Mississippi River on one side and upland cover on the other sides. Various levels of protection from floods exist at the different study sites. Some of the sites are hydraulically connected with the Mississippi River via open water channels and others are separated from the river by levees. Some of the sites receive upland run-off and others are protected from run-off by upland levees or forest zones. Refer to Table 2 for an outline of the land features present at each study site.

Table 1. Location information for the study sites, Mark Twain National Wildlife Refuge contaminants study, 1992.

| Study Site | Pool | River Mile | County | State |
|------------------------|-------------|-------------------|---------------|--------------|
| Big Timber Division | 17 | 443 | Louisa | Iowa |
| Keithsburg Division | 18 | 428 | Mercer | |
| Skunk Slough | 19 | 396.4 | Lee | Iowa |
| Gardner Division | 20 | 332.6 | Adams | |
| Clarence Cannon Refuge | 25 | 260.5 | Pike | Missouri |
| Batchtown Division | 25 | 246 | Calhoun | Illinois |

Table 2. Levee¹ and general land cover information for the study sites, Mark Twain National Wildlife Refuge, 1992.

| Study Site and Acres | River Levee | Upland Levee | Adjacent Land Cover |
|---|--------------------|---------------------|----------------------------|
| Big Timber Division (3,375 acres) | No | Yes | Agricultural |
| Keithsburg Division (1,400 acres) | Yes | No | Agricultural |
| Skunk Slough (1,500 acres) | No | No | Forest |
| Gardner Division (6,000 acres) | No | No | Forest |
| Clarence Cannon Refuge (3,750 acres) | Yes | Yes | Agricultural |
| Batchtown Division (2,250 acres) | No | No | Agricultural |

Backwater is not hydraulically connected to rivers and streams during normal stage

Sampling Locations

We targeted locations in each study site where fine grain sediments are likely deposited. These optimal sampling locations were established at tributary inlets and non-vegetated basins within each site. Several optimal sampling locations were assessed at each study site depending on the size of the backwater and accessibility.

This strategy should identify maximum sediment contaminant concentrations for each site because inorganic contaminants are associated to a greater degree with fine grain sediments. This is because contaminants that are released into surface water generally bind more readily to silt, clay and organic matter. Waterborne contaminated sediments are transported as suspended solids in rivers and streams and are deposited in slack water areas where they settle out of the water column under low flow conditions.



Methods

The Sediment Quality Triad approach (Long and Chapman 1985; Chapman 1986) was attempted to identify sites with contaminant problems and show if there were ecological impacts from pollution. The Triad approach used three assessment methods. The assessment methods included sediment chemistry, evaluation of infaunal benthos diversity and sediment toxicity testing. The relationships of the results from the assessment methods were used to characterize ecological impacts. Adverse impacts may include low benthos diversity, mortality in toxicity tests and elevated chemical concentrations.

Sediment Quality

Sediments were collected with a standard Ekman dredge with a pole handle. Two sediment grab samples at ten feet apart were taken at each location. The dredge was inserted, closed and raised for inspection. The contents were emptied into a stainless steel bowl if the dredge was at least three quarters full. This included between 13 and 19 centimeters of the sediment column. If it was not a complete grab, another grab was attempted approximately six feet in any direction from the last attempt. The material was gently mixed with a stainless steel spoon and portions were scooped into chemically clean containers for analyses. The samples were chilled in a cooler with blue ice or wet ice. The samples were mailed or transported to the office for storage in a refrigerator or standard chest freezer depending on the type of analyses to be performed. The samples were forwarded to contract laboratories for analysis according to the Chemical Data Acquisition Plan (Table 3). Both grab samples from each location were analyzed for heavy metals and texture data. The first of the two grabs was also analyzed for ammonia. Seventeen archived samples were later analyzed for phosphates.

Table 3. Chemical data acquisition plan for the Mark Twain National Wildlife Refuge contaminants study, 1992.

| Analyte | Analytical Method | Quantitation Limit |
|-------------------------|----------------------------------|-----------------------|
| Arsenic | Graphite furnace AA ¹ | 0.5 µg/g ² |
| Cadmium | Graphite furnace AA | 0.2 µg/g |
| Chromium | ICP ³ | 1.0 µg/g |
| Copper | ICP | 1.0 µg/g |
| Lead | Graphite furnace AA | 5.0 µg/g |
| Mercury | Cold vapor AA | 0.1 µg/g |
| Nickel | ICP | 5.0 µg/g |
| Selenium | Graphite furnace AA | 1.0 µg/g |
| Zinc | ICP | 5.0 µg/g |
| Total organic carbon | Coulometrically | |
| Grain size distribution | Sieve weight | |
| Ammonia-nitrogen | Phenate Method | 0.1 µg/g |
| Phosphate-phosphorus | Automated Ascorbic Acid Method | 0.1 µg/g |

Atomic Absorption Spectroscopy

² Micrograms per Gram

³ Inductively Coupled Plasma Emission Spectroscopy

Infaunal Macroinvertebrates

Sediment grab samples were collected as described above. Care was taken not to dredge the same spot from which the sediment chemistry samples were collected. The contents from the grab were emptied into plastic bags and labeled for storage. In the field, samples were maintained chilled in a cooler with blue ice or wet ice. The samples were mailed or transported to the office laboratory for storage in a standard chest freezer.

A notation was made on the field data sheet if burrowing mayflies or fingernail clams were observed during field handling (*eg.* stirring or spooning sediment into sample containers) of the sediment samples. The organisms were easy to detect if they were present above low numbers because of their size and movement. The fingernail clams were not active directly after collection, but were large enough by May for easy detection. Mayflies were easy to detect because they constantly moved about and fanned their tail and gill structures.

At least two frozen samples from each site were randomly selected and processed for macroinvertebrates. The contents of each plastic bag were completely emptied into a benthos bucket (Wildco Company) with a mesh size of 0.595 millimeters. The sediment was washed from the debris and organisms with tap water through a garden hose. The material trapped by the sieve was backwashed into a white enamel pan.

Fingernail clams with articulated valves, burrowing mayfly nymphs and other recognizable taxonomic groups present in the material were enumerated and the sample discarded.

Toxicity Testing

Sediment grab samples were collected as described above. Care was taken not to dredge the same spots from the sediment chemistry and macroinvertebrate samples were collected. The samples were maintained in a cooler with blue ice or wet ice. The samples were forwarded to the office laboratory for storage in a standard chest freezer for Microtox[®] testing and in a refrigerator for the sediment elutriate toxicity test.

The Microtox[®] solid phase sediment assay test was performed on sediments from all of the sites to rank the relative toxicity between the sites. Elutriate toxicity tests were performed on sediments from one site (Keithsburg Division) to help interpret the results of the Microtox[®] test.

Microtox[®] Test

Microtox[®] assays were performed in duplicate on at least two samples randomly selected from each site. The assays were completed at the Rock Island Field Office laboratory. The Microbics version 6.3 detailed solid-phase test was used (Microbics, 1989 *et. seq.*). Room temperature, color and turbidity were monitored.

Sediment Elutriate Test

The University of Iowa Hygienic Laboratory performed the elutriate toxicity tests. Sediments from Keithsburg Division were tested because metal and ammonia concentrations were elevated for the study area. The toxicity tests were duplicate 96- hour static acute bioassays. A 200 milliliter volume of sediment was thoroughly mixed with purified laboratory water at a ratio of 1:4. The elutriate was allowed to settle for about 60 hours before the introduction of larval fathead minnows (*Pimephales promelas*).

The bioassay water was aerated and the fish were fed brine shrimp on days three and five. The bioassay water was monitored for temperature, pH, dissolved oxygen, ammonia. Un-ionized ammonia concentrations were calculated for each bioassay vessel.

Water Quality

Selected water quality measurements were taken at approximately 0.3 meters (one foot) below the surface and approximately 0.3 meters above the substrate if greater than three meters deep. Table 4 lists the water quality parameters and instruments used.

Table 4. Water quality parameters and instruments used for the Mark Twain National Wildlife Refuge contaminants study, 1992.

| Parameter | Instrument Units | Instrument | Precision |
|------------------|--------------------|---------------------|--------------|
| Depth | meters | Marked pole | |
| Temperature | degrees celsius | YSI Meter Model 51 | |
| Dissolved oxygen | milligrams/liter | YSI Meter Model 51B | 25% of scale |
| Conductivity | μ S/centimeter | YSI Meter Model 33 | 2% of scale |

Data Analyses

Statistical Analysis

Descriptive statistics of the contaminants data were calculated using Excel version 4.0 (Microsoft Corporation). Descriptive statistics included arithmetic mean, standard deviation, minimum and maximum values. Correlation coefficients (*r*) for the chemistry and texture data were calculated using Excel.

The results of the Microtox[®] assays were analyzed using Microtox[®] software version 6.3 (Microbics Corporation) to determine the EC₅₀ which is the concentration at which effective response by the bacteria was reduced by 50%.

GIS Analysis

The spatial relationships of the contaminants and environmental data were analyzed using Environmental Planning and Programming Language version 7.0 (EPPL7) (State of Minnesota, Land Management Information Center, St. Paul, MN).

The environmental data included land cover, hydrography and industrial wastewater discharge points for MTNWR. The land cover was available from UMR floodplain satellite imagery attributed by the National Biological Survey's Environmental Management Technical Center (Onalaska, WI). The wastewater discharge locations were gathered from state inventories of National Point Discharge Elimination System permits (NPDES).

Quality Assurance and Quality Control

Field data sheets were completed at the time of sampling and included information on water quality, aquatic plant growth, substrate condition, date, time, weather and collector's name.

The position of the sampling locations were plotted on detailed refuge maps. The points were later entered on the GIS base map and Universal Transverse Mercator coordinates (UTM) were obtained for the sampling locations.

The instrument probes and collection gear were thoroughly rinsed with river water, then acetone (if appropriate for the gear) and de-ionized water between uses.

The dissolved oxygen meter was calibrated by the air calibration technique. The precision of the field equipment are listed in Table 4.

The contract analytical laboratories conducted quality assurance and quality control tests with six randomly selected samples and reference media. The tests included procedural blank samples, replicate tests, testing reference materials and spike recovery analysis.

Control and reference toxicant bioassays were conducted with the elutriate toxicity tests.



Results

Sediment Quality

Sediment Texture

The dominant grain sizes at the sampling stations were silt and clay. Sediment total organic carbon content concentrations (TOC) were less than five percent with Keithsburg Division samples in the high part of this range. Average texture data for the study sites are outlined in Table 5.

One sampling location out of the total of 85 had a high percentage of sand (88 percent). This location was in a small bay of Big Timber Division adjacent to upland sandy soils. The contaminant data from this location was excluded from descriptive and correlation analyses because inorganic pollution is generally not associated with substrates of quartz sand (Brannon *et al* 1976). The texture data from each sampling station are listed on copies of the original spreadsheets in Appendix B.

Table 5. Average sediment texture data for study sites, Mark Twain National Wildlife Refuge, 1992.

| Study Site | %Organic | %Clay | %Silt | %Sand |
|------------------------|----------|-------|-------|-------|
| Skunk Slough | 2.4 | 45.85 | 49.07 | |
| Big Timber Division | 1.95 | 33.61 | 51.69 | |
| Keithsburg Division | 3.54 | 36.09 | 52.13 | 11.79 |
| Gardner Division | 1.36 | 17.68 | 59.18 | 23.14 |
| Clarence Cannon Refuge | 2.52 | 47.31 | 45.72 | |
| Batchtown Division | 1.93 | 39.15 | 53.05 | 7.38 |

Sediment Chemistry - Metals

The average sediment concentrations of arsenic and heavy metals for the study area are listed in Table 6. Selenium and mercury were not detected. The mean metal concentrations for the study area except for zinc were within background concentrations for soils and aquatic sediments in Illinois (Table 7). Zinc was 1.5 times greater than average background concentrations for soil and 1.7 times greater for aquatic sediments in Illinois. The correlation of arsenic and heavy metals with grain size distribution and TOC content are provided in Table 8. The best associations were copper with percent clay ($r=0.77$) and cadmium with TOC ($r=0.63$). The average concentrations of arsenic and heavy metals varied slightly between the sampling sites with concentrations exceeding background at some sampling locations (Table 9). The chemistry data from each sampling station are listed in Appendix B.

Table 6. Mean, minimum and maximum arsenic and heavy metal concentrations in sediments (micrograms per gram, dry weight, $n=83$) for the study area, Mark Twain National Wildlife Refuge, 1992.

| Analyte | Mean ($n=83$) | Standard Deviation | Minimum | Maximum |
|----------|-----------------|--------------------|-----------------|---------|
| Arsenic | 5.32 | 1.61 | 0.5 | 8.18 |
| Cadmium | 0.45 | 0.17 | ND ¹ | 0.77 |
| Chromium | 18.53 | 9.02 | 3.69 | 50.52 |
| Copper | 21.05 | 6.01 | 7.70 | 34.65 |
| Nickel | 21.36 | 8.15 | ND | 44.30 |
| Lead | 21.19 | 7.07 | 7.82 | 51.92 |
| Zinc | 83.62 | 29.36 | 16.00 | 145.6 |

ND = not detected above quantitation limit for this analyte

Table 7. Average arsenic and heavy metal concentrations in sediments (micrograms per gram, dry weight) for the study area with comparison data, Mark Twain National Wildlife Refuge, 1992.

| Analyte | Study Area | | Soils | | Background Sediments | |
|----------|------------|-------|-----------------|-------------------|--------------------------|-------------------------|
| | Mean | Max | MN ¹ | U.S. ² | Great Lakes ³ | IL Streams ⁴ |
| Arsenic | 5.32 | 8.18 | - | | <3 | <8 |
| Cadmium | 0.45 | 0.77 | 0.3 | | | <0.5 |
| Chromium | 18.53 | 50.52 | 43 | 53 | <25 | <16 |
| Copper | 21.05 | 34.65 | 26 | 25 | <25 | <38 |
| Nickel | 21.36 | 44.30 | 21 | 20 | <20 | |
| Lead | 21.19 | 51.92 | <25 | 20 | <40 | <28 |
| Zinc | 83.62 | 145.6 | 54 | 54 | <90 | <80 |

Average Minnesota soil profiles (n=24) (Baily and Rada 1984)

Average elemental concentrations in surficial materials (Schacklette *et al* 1971)

Average concentrations for non-polluted harbor sediments in the Great Lakes (n=260, USEPA 1977).

Non-elevated stream sediments in Illinois (n=79, IEPA 1984).

Table 8. Correlation¹ of arsenic and heavy metals with total organic carbon content and percent clay for the study area, Mark Twain National Wildlife Refuge, 1992.

| | As ² | Cd | Cr | Cu | Ni | Pb | Zn |
|-------------------|-----------------|------|------|------|------|------|------|
| %TOC ³ | 0.20 | 0.63 | 0.15 | 0.47 | 0.18 | 0.26 | 0.29 |
| %Clay | 0.33 | 0.53 | 0.28 | 0.77 | 0.47 | 0.50 | 0.5 |

The correlation coefficient (*r*) is a measure of the closeness of the relationship between two variables, *r* = 1 indicates a perfect relationship

Arsenic, cadmium, chromium, copper, nickel, lead and zinc, respectively

Percent total organic carbon content

Table 9. Average sediment arsenic and heavy metal concentrations (micrograms per gram, dry weight) for the study sites, Mark Twain National Wildlife Refuge, 1992.

| Study Site | As ¹ | Cd | Cr | Cu | Ni | Pb | Zn |
|---------------------------------|-----------------|------|-------|-------|-------|-------|--------|
| Skunk Slough (n = 6) | 6.60 | 0.43 | 17.12 | 28.94 | 21.28 | 30.30 | 92.73 |
| Big Timber Division (n = 16) | 5.39 | 0.42 | 21.58 | 21.51 | 25.05 | 26.45 | 108.11 |
| Keithsburg Division (n = 14) | 6.10 | 0.57 | 20.66 | 22.52 | 21.07 | 21.61 | 89.53 |
| Gardner Division (n = 8) | 4.35 | 0.38 | 10.58 | 11.45 | 9.87 | 11.76 | 45.92 |
| Clarence Cannon Refuge (n = 15) | 3.71 | 0.35 | 13.05 | 20.09 | 17.21 | 17.87 | 66.10 |
| Batchtown Division (n = 24) | 5.83 | 0.49 | 21.67 | 21.71 | 26.52 | 20.39 | 85.08 |

Arsenic, cadmium, chromium, copper, nickel, lead and zinc, respectively

Sediment Chemistry - Nutrients

The average concentrations of sediment nutrients from the study sites are listed in Table 10. Sediment ammonia concentrations were slightly to very elevated as compared to background sediment chemistry data (Table 10). Keithsburg Division had the highest concentrations in the study area of sediment ammonia and phosphates (average total ammonia-nitrogen = 231.43 micrograms/gram; phosphate = 2000.0 micrograms/gram). Ammonia concentrations had good correlation with TOC ($r=0.75$). The chemistry data from each sampling station are listed in Appendix B.

One sampling station at the south end of Keithsburg Division had a bulk sediment ammonia concentration of 1400 micrograms per gram, dry weight. The results from this sampling station were excluded from the calculations for the descriptive and graphical statistics because it was so much higher than the ammonia data from the other seven sampling stations at Keithsburg Division.

Table 10. Average sediment ammonia and phosphate concentrations (micrograms per gram, dry weight) for the study area with comparison data, Mark Twain National Wildlife Refuge, 1992.

| Sampling location | Ammonia-nitrogen | Phosphate |
|---------------------------|------------------|------------------|
| Skunk Slough | 81.66 (n = 3) | 1100.0 (n = 1) |
| Big Timber Division | 103.00 (n = 7) | 795.0 (n = 4) |
| Keithsburg Division | 231.43 (n = 7) | 2000.0 (n = 2) |
| Gardner Division | samples lost | samples lost |
| Clarence Cannon Refuge | 102.40 (n = 5) | 742.5 (n = 4) |
| Batchtown Division | 83.30 (n = 6) | 846.7 (n = 6) |
| Non-polluted ¹ | 75.00 (n = 260) | 1369.0 (n = 260) |

The means were used to classify non-polluted aquatic sediments from Great Lakes harbors between 1974 and 1975 (USEPA 1977).

Infaunal Macroinvertebrates

Midge larvae (families Chironomidae and Heleidae) and aquatic worms (class Oligochaeta) were observed throughout most of the study area.

In addition to midges and aquatic worms, abundant burrowing mayfly nymphs together with fingernail clams were collected at the reference site (Skunk Slough) and abundant burrowing mayflies were collected at Gardner Division. Infaunal macroinvertebrates were poorly represented in the sediment samples from Keithsburg Division.

The number and types of organisms collected at the study sites are outlined in Table 11. The macroinvertebrate data from each sampling station are listed in Appendix C.

Table 11. Average number and the taxa of infaunal macroinvertebrates per grab sample for locations at the study sites at Mark Twain National Wildlife Refuge, 1992.

| Study Site | Midge larvae Chironomidae | Fingernail Clams Spaeriidae | Burrowing Mayflies <i>Hexagenia</i> |
|------------------------------|------------------------------|--------------------------------|--|
| Skunk Slough (n = 4) | 2.25 | 3.25 | 3.5 |
| Big Timber Division (n = 10) | 3.7 | 0 | 0.1 |
| Keithsburg Division (n = 5) | 0.2 | 0 | 0 |
| Gardner Division (n = 2) | 8.5 | 0.5 | 3 |
| Batchtown Division (n = 3) | 8.7 | 0.3 | 0 |

Toxicity Testing

Microtox® Test

The average effective concentrations (EC_{50} s) for the study sites are listed in Table 12. The EC_{50} value is the concentration at which 50 percent of the organisms exhibited a response for each of the Microtox® assays. The lower EC_{50} values are considered to represent a more contaminated media, for it takes less material for a response. There was a good correlation with test results and sand grain size ($r = 0.97$).

The results suggest that the sediments from some of the sampling locations produced a response by the test organisms and the relative magnitude of the response as measured with this organism varied greatly. Mean EC_{50} value from Skunk Slough was measured as the most potent compared to the other study sites. Copies of the Microtox® program printouts are in Appendix D.

Table 12. Average Microtox[®] effective concentrations (EC₅₀), micrograms per gram) for the study sites at Mark Twain National Wildlife Refuge, 1992.

| Study Site | Mean EC ₅₀ | SD | Minimum | Maximum |
|------------------------|-----------------------|---------|---------|-----------|
| Skunk Slough | 664.84 (n = 4) | 213.28 | 334.85 | 2659.3 |
| Big Timber Division | 3128.72 (n = 4) | 2359.88 | 1417.03 | 6581.97 |
| Keithsburg Division | 1454.83 (n = 4) | 358.33 | 1011.78 | 1881.13 |
| Gardner Division | 6574.05 (n = 4) | 8593.05 | 544.98 | 16,236.97 |
| Clarence Cannon Refuge | 2267.16 (n = 7) | 975.58 | 842.85 | 3441.76 |
| Batchtown Division | 1228.42 (n = 8) | 1234.82 | 343.2 | 3788.81 |

Sediment Elutriate Test

There was zero percent mortality in July and up to 20 percent in September of the larval fish used in the elutriate tests at Keithsburg Division (Table 13). Bioassay water quality in the test vessels were normal except for un-ionized ammonia (see data in Table 13). The concentrations of un-ionized ammonia in four out of the six tests at the 48 hour time exceeded lethal concentrations for fathead minnows (0.70 milligrams per liter-mg/L, 48 hours LC₅₀) (USEPA 1985). Copies of the laboratory results for each test are in Appendix E.

Table 13. Results for the sediment elutriate toxicity tests for the study area, Mark Twain National Wildlife Refuge, 1992.

| Test | Mortality ¹ | | Un-ionized Ammonia (milligrams per liter) | | |
|-----------|------------------------|---------|---|----------|----------|
| | Sample | Control | Initial | 48 Hours | 96 Hours |
| 7-14-92 A | 0/20 | 0/20 | 0.12 | 0.43 | 0.16 |
| 7-14-92 B | 0/20 | 1/20 | 0.12 | 0.40 | 0.20 |
| 9-17-92 A | 4/20 | 0/20 | 0.18 | 0.92 | 0.34 |
| 9-17-92 B | 1/20 | 0/20 | 0.19 | 1.2 | 0.34 |
| 9-17-92 C | 4/20 | 0/20 | 0.27 | 1.0 | 0.33 |
| 9-17-92 D | 4/20 | 0/20 | 0.22 | 1.0 | 0.35 |

¹ Mortality = number dead / number tested

Water Quality

Water quality parameters measured for this project were similar throughout the study area except for dissolved oxygen (DO). Dissolved oxygen was good during most sampling trips except for later trips to Keithsburg Division. Dissolved oxygen levels were low at some locations during July and September at Keithsburg Division. The ranges for the water quality data are summarized in Table 14. The water quality data from each of the sampling locations are listed in Appendix F.

Table 14. The ranges of water quality data for the study sites at Mark Twain National Wildlife Refuge, 1992.

| Study Site | Month | Depth (Meters) | Temperature (Celsius) | Conductivity (μ S/cm) | Dissolved Oxygen (Milligrams/liter) | |
|------------------------|---------|-------------------|--------------------------|-------------------------------|--|----------|
| | | | | | Top | Bottom |
| Skunk Slough | Jun | 1.0-2.5 | 26.0-27.0 | 440-470 | 6.2-10.0 | |
| Big Timber Division | May/Jun | 0.5-5.5 | 23.0-25.0 | 310-400 | 7.5-14.0 | 5.8-6.2 |
| Keithsburg Division | May | 2.75-5.5 | 20.2-22.5 | 310-340 | 5.2-15.0 | 3.4-12.0 |
| | Jul | 3.0-4.0 | 25.5-26.0 | 280-315 | 4.1-4.4 | 0.5-3.6 |
| | Sep | 2.5 | 19.0-20.0 | 350-470 | 2.0-5.7 | 0.6-4.0 |
| Gardner Division | May | 1.5-1.75 | 19.5-20.5 | 310-455 | 8.1-9.2 | |
| Clarence Cannon Refuge | May | 0-11.0 | 17.0-19.0 | 190-430 | 7.5-12.0 | |
| Batchtown Division | May | 1.5-4.5 | 20.5-23.0 | 285-335 | 5.4-15.0 | 11-15.0 |

Quality Assurance and Quality Control

Quality assurance performance results for the sediment analyses conducted by U.S. Fish and Wildlife Service contract laboratories were accepted by the Service's quality control office at the Patuxant Analytical Control Facility (PACF). There were slight differences in the replicate test results. The results of the internal laboratory replicate, spike and procedural blank analyses are provided in Appendix G. The contract laboratories' analytical results and original field data sheets are held at the Rock Island Field Office.

Discussion

Sediment Quality

Metals

Mean concentrations of arsenic and heavy metal contaminants were not at levels of concern. Skunk Slough was a site with above average heavy metal concentrations for the study area and both pollution sensitive species of infaunal macroinvertebrates were present in abundance. Skunk Slough was also the site demonstrating an effective response with the Microtox[®] toxicity test. This relationship provides evidence that these concentrations of arsenic and heavy metals may not be limiting benthic macroinvertebrate production in Refuge backwaters and that the Microtox[®] results are inconclusive.

Nutrients

Sediment ammonia concentrations between the low and high ends of the range for the study area may be limiting aquatic life in MTNWR backwaters. Ammonia concentrations in Skunk Slough were near the low end of the range for the study area and that backwater supported abundant pollution-sensitive macroinvertebrates. Sediment ammonia concentrations at Keithsburg Division were at the high end of the range for the study area and infaunal macroinvertebrates were poorly represented there. Sediment ammonia concentrations at Keithsburg Division reached toxic levels for fish as indicated by the elutriate toxicity tests.

Ammonia that is found in the substrate is mostly bound to surrounding fine grain particles in the form of ammonium (NH_4^+). A portion of the ammonia is in solution as un-ionized ammonia (NH_3). The un-ionized ammonia accumulates within the sediment

pore water and is released at the substrate-water interface. Un-ionized ammonia is toxic to aquatic organisms at very low concentrations (USEPA 1986). Un-ionized ammonia is generally less toxic to invertebrates (lethal concentration for midge larvae, *Chironomus* sp., is $LC_{50} = 6.6$ mg/L for 96 hours] as compared to fish (lethal concentration for bluegill, *Lepomis* sp., is $LC_{50} = 1.75$ mg/L for 96 hours] (USEPA 1976 and 1985). The fraction of un-ionized ammonia is calculated using temperature and pH.

Phosphates in sediments are not typically toxic to aquatic animals (USEPA 1986). The significance of high phosphates is the effect of causing nuisance aquatic plant blooms and the resulting build-up of organic matter (USEPA 1986). Aquatic plant blooms can limit dissolved oxygen from excessive plant respiration relative to oxygen production through photosynthesis and by the microbial decay of dead plant tissue.

Potential Sources - Nutrients

Keithsburg Division had higher sediment nutrient concentrations and higher organic content compared to the other study sites in MTNWR (Table 10). There are likely three sources for nutrients at Keithsburg Division. The first source is ammonia produced in lake sediments from decaying organic matter. Agricultural fertilizer run-off is an important source for nitrogen and phosphorus compounds. The third source is from municipal and industrial wastewater point discharges.

Organic matter that is deposited in the substrate of aquatic and wetland systems is decomposed by bacteria which produce ammonia as a by-product. The ammonia is converted first to nitrites then to nitrates by bacteria (Rand and Petrocelli 1985). The amount of ammonia production depends on the volume and quality of the organic matter and overlying water quality conditions (Rand and Petrocelli 1985). The conversion to nitrate

can consume significant amounts of dissolved oxygen from the water column, especially during the winter after a season of ammonia production (Knowles and Lean 1987).

Potential Pathways - Nutrients

Analysis of the spatial data for Keithsburg Division demonstrated the use of GIS for contaminants investigations and helped identify contaminant pathways. Figure 3 is GIS map product to display the land cover around Keithsburg Division. Overlay data for the spatial analysis included wastewater discharge points and tributary rivers and streams.

There was only one wastewater discharge point near Keithsburg Division. It is the discharge into the Mississippi River for the municipal sewage treatment plant for New Boston, IL. This source is separated from the Keithsburg Division backwater by the river levee. Surface water from the Mississippi River enters the backwater only during flood stages. The 1993 flood on the Mississippi River overtopped the river levee for much of the summer and created a scour route to Pope Creek from Keithsburg Division. Solids and contaminants transported in the flood water may have changed sediment quality at Keithsburg Division.

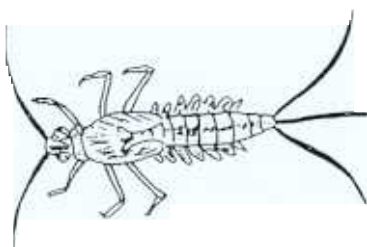
There are no upland levees at Keithsburg Division. The upland cover adjoining Keithsburg Division is agricultural. Surface runoff from adjacent agricultural fields may carry contaminants to the site. The primary sources of surface water for Keithsburg Division are the Edwards River to the north, small un-named tributaries at the north boundary and Pope Creek to the south. The Edwards River and Pope Creek are slightly to moderately impaired from suspended solids, nutrients and habitat modifications (IEPA 1994). Inputs from the Edwards River and Pope Creek may lead to poor water quality at Keithsburg.

Infaunal Macroinvertebrates

In-lake production of ammonia from organic enrichment likely limits benthic communities at Keithsburg Division backwaters. Infaunal macroinvertebrates were sparse at Keithsburg Division in May of 1992. Poor sediment quality and eutrophication were evidenced at Keithsburg Division by the abundant aquatic plant growth, low dissolved oxygen, high organic content and sediment ammonia toxicity.

Ammonia toxicity has been blamed for the decline of fingernail clams in the Illinois River and the Mississippi River. Highly eutrophic backwater lakes should shift from having diverse macroinvertebrate populations to populations of only a few pollution-tolerant organisms. Sustaining macroinvertebrate production in Mississippi River backwaters is important because they represent critically important food items for migratory birds and fish. Fingernail clams may be locally important to waterfowl.

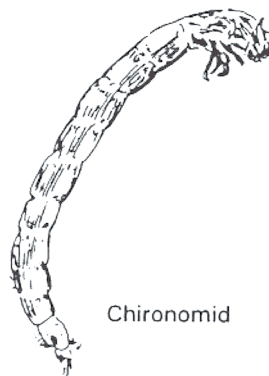
The absence of fingernail clams and burrowing mayflies in backwater sites may be explained by reasons other than sediment pollution. Macroinvertebrate communities may have a random distribution and their absence does not necessarily indicate poor quality habitat. Backwaters with high sediment organic content may be unsuitable for burrowing mayfly nymphs. However, fingernail clams are typically found in shallow, rich backwaters along the Upper Mississippi River.



Mayfly nymph



Fingernail clam



Chironomid



Oligochaete

Figure 3. Infaunal macroinvertebrate illustrations.

Toxicity Testing

Microtox® Test

The Microtox® assay was used to test for toxicity of backwater sediments. This system is relatively new, inexpensive to run and easy to use. The Microtox® system measures luminescence of the marine bacterium *Photobacterium phosphoreum*. Inhibition of this luminescence is considered a toxic response. Results of the Microtox® assay compare to other standard freshwater toxicity tests using fish and cladocerans (Kaiser and Palabrica 1991). Natural toxins like sulfur in aquatic sediments are also lethal to this species of bacteria (Jacobs *et al* 1992). *P. phosphoreum* are not sensitive to ammonia (Ankley *et al* 1990). The heavy metals and free sulfur from sulfide compounds commonly found in the deeper and anoxic part of the sediment column may have contributed to the effect measured using *P. phosphoreum* for this study.

The results also demonstrated that fine grain sediments caused a greater response than coarse grain sediments. Coarse grain sediments like sand and large silt sizes do not have the same capacity to bind contaminants and generally do not hold high concentrations of inorganic pollutants (USEPA 1976). Fine grain sediments increased the turbidity of the sample and may have affected the measurement of the light output.

The Microtox® test did not compare well to the sediment elutriate test because of the presence of ammonia. The Microtox® organism is not sensitive to ammonia and fish species are very sensitive to ammonia. Overall, the Microtox® test did not adequately analyze the toxicity of backwater sediments because of the role of ammonia toxicity and turbidity interferences.

Sediment Elutriate Test

Ammonia concentrations were high in the bioassay water for the elutriate toxicity tests at Keithsburg Division. Sediment bound ammonia is easily released in the water column when mixed (Brannon *et al*/ 1978). Heavy metals bound to sediments tend not to be released at full concentrations into the water column under neutral and toxic conditions when mixed (Brannon *et al*/ 1978).

Sediment Quality Triad

The data from the Microtox® tests may not be used for the Sediment Quality Triad analysis because the Triad is an effects-based approach incorporating measures of chemistry, benthos diversity and toxicity (Chapman *et al*/ 1992). The Triad analysis depends on satisfactory toxicity test results to rank degradation between study sites (Chapman *et al*/ 1991).

The elutriate toxicity tests for Keithsburg Division were intended to validate the Microtox® tests. The elutriate tests were used with benthos data in the Triad approach for this site. There was evidence that the toxicity test results were related to ammonia contamination. This may explain the poor benthos diversity observed at Keithsburg Division.

Water Quality

Water quality was measured only during sediment sampling trips to provide information to help interpret of the sediment chemistry data. The levels of dissolved oxygen at Keithsburg Division were at times below criteria intended to protect native fish communities (criteria = 5 mg DO/L).

Dissolved oxygen levels may vary greatly during the day and throughout the season in backwater lakes with high primary production (Owens and Crumpton 1993). Refer to Figure 5 for an illustration of dissolved oxygen cycles in a productive backwater lake of the Upper Mississippi River. The lowest oxygen levels typically occur near daybreak due to overnight respiration of phytoplankton and other aquatic plants. Supersaturated oxygen conditions typically occur in the afternoon as a result of photosynthesis.

The importance of the low dissolved oxygen observations is that it supports the expected affects of oxygen demand by high plant respiration and organic matter decay related to nutrient enrichment found at Keithsburg Division.

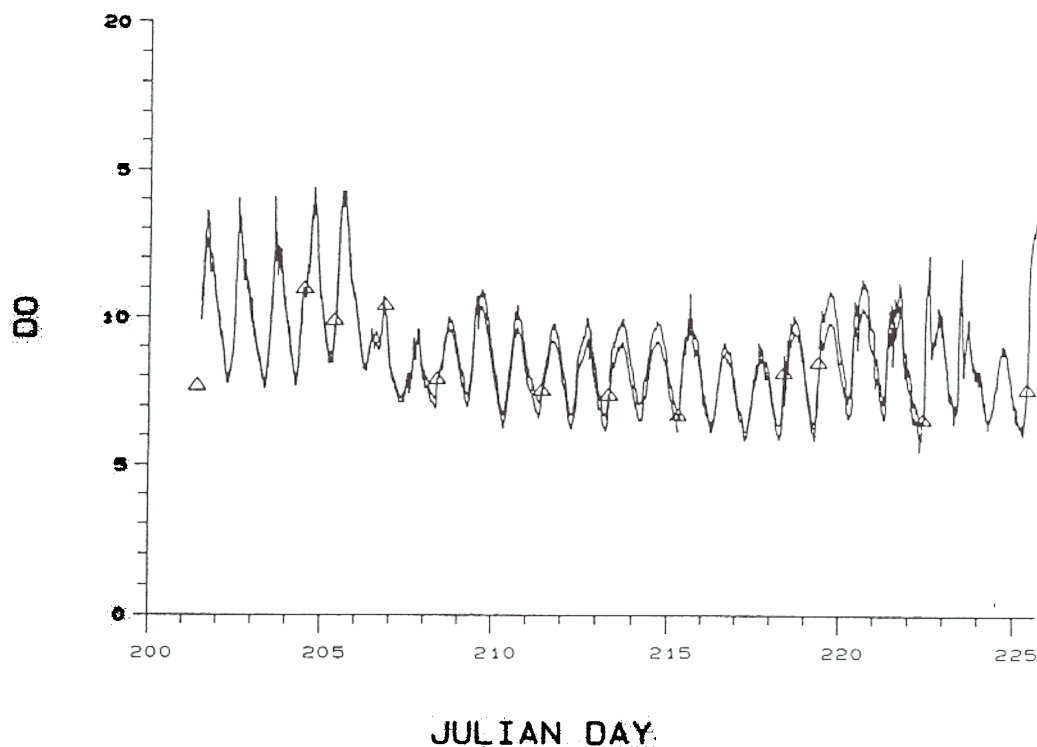


Figure 5. Example of dissolved oxygen levels (DO in parts per million) at a productive backwater lake along the Upper Mississippi River (taken from Owens and Crumpton 1993).

Conclusions

Prior to the greater than one hundred year flood in 1993, MTNWR backwater sites along the UMR were not grossly polluted with organic pollutants and heavy metals (Young 1991 and this study).

Skunk Slough in Pool 19 was used as a reference site for this study because it supported abundant pollution-sensitive macroinvertebrate species. Skunk Slough had above average concentrations of sediment heavy metals and low sediment ammonia concentrations for the study area.

In contrast to Skunk Slough, ammonia toxicity, nutrient and organic enrichment were found in the backwater habitats of Keithsburg Division. Keithsburg Division also had above average sediment heavy metal concentrations. This site had the highest ammonia concentrations in the study area. There was some mortality in the elutriate toxicity tests conducted later in the season and infaunal macroinvertebrates were poorly represented at Keithsburg Division. GIS analysis indicated that the sources and pathways for contaminants at Keithsburg Division were related to non-point source pollution versus point source pollution.

The methods used for the Sediment Quality Triad approach provided worthwhile data to help describe contaminant-related problems at Keithsburg Division. However, the Microtox[®] toxicity tests from throughout the study area were inconclusive. The Microtox[®] test results were not used with the chemistry and benthos data to help describe sediment quality at the other study sites according to the Sediment Quality Triad approach. We found that the Microtox[®] test results did not relate to benthos diversity and sediment toxicity because of the role of ammonia and turbidity in the test samples.

Recommendations

Management Recommendation

We recommend that the Division of Environmental Contaminants and Division of Refuges formulate strategies to help manage non-point source pollution in backwater habitats of MTNWR. Non-point source pollution includes increased sedimentation and nutrient loading. Nutrient enrichment promotes ammonia production and leads to cultural eutrophication. There are many nutrient management strategies available to reduce the effects from non-point source pollution. These strategies may be grouped into two categories and include on-refuge and off-refuge activities.

On-refuge strategies include diversion of inputs and management of refuge sections as chemical treatment wetlands. Treatment wetland technology is used for nutrient management at municipal sewage treatment plants. Treatment wetlands can assimilate some nutrients and trap solids.

Off-refuge strategies include increasing riparian buffer zones, reducing run-off potential and instituting integrated pest management programs in the watershed.

Many of these strategies may be incorporated into habitat restoration projects by the Environmental Management Program and Farm Bill programs.

No one pollution management method will remove all types of nutrients and filter chemicals continuously. Integrating strategies specific to each site will help protect migratory birds and fish resources. These strategies should improve water quality, generate diverse aquatic macrophyte communities and optimize production of forage resources.

Research Recommendations

- 1 Catalog the sediment quality data into the GIS database for MTNWR. The sediment quality database currently under construction by the National Biological Survey for the UMR may be another appropriate repository for these data.**

The 1989 and 1992 studies at MTNWR provide baseline data for management purposes and pre-spill conditions for a large U.S. Fish and Wildlife Service refuge. These data are needed for natural areas located along transportation routes for hazardous materials. The transportation routes in MTNWR include barge traffic on the UMR, gas pipelines at Keithsburg Division and various road and railroad bridges over tributaries leading to MTNWR units. The baseline data are available for natural resource damage assessments in the event of a spill.

- 2. Repeat biomonitoring at MTNWR every five years to evaluate long-term trends in pollution.**

The Sediment Quality Triad with modifications is the recommended approach to assess trends in pollution. Many different types of contaminants are deposited in the substrate and sediment quality data provided a status report on contamination to individual backwater systems. Using the Triad approach provided useful ecological data for resource managers. The biomonitoring data are also needed by three other programs under development by the U.S Fish and Wildlife Service. The programs include long-term biomonitoring of refuges (Biomonitoring Environmental Status and Trends - BEST), the migratory bird strategy for the Upper Mississippi River and ecosystem management plans for the Upper Mississippi River.

The modifications to the Sediment Quality Triad approach include changing to ammonia sensitive toxicity tests (see Dillon and Ross 1990) and designing statistically defensible methods.

3. Identify non-point source pollution problems for MTNWR and develop management strategies.

We used the nutrient pollution data and GIS results from this project as an indication of risk from agricultural chemical pollution such as herbicides and insecticides. A risk assessment funded by the Divisions of Refuges and Ecological Services was initiated at Keithsburg Division in 1993 and continues in 1995. The risk assessment will evaluate all of the pathways to the site and provide specific management alternatives to reduce the adverse effects from non-point source pollution.

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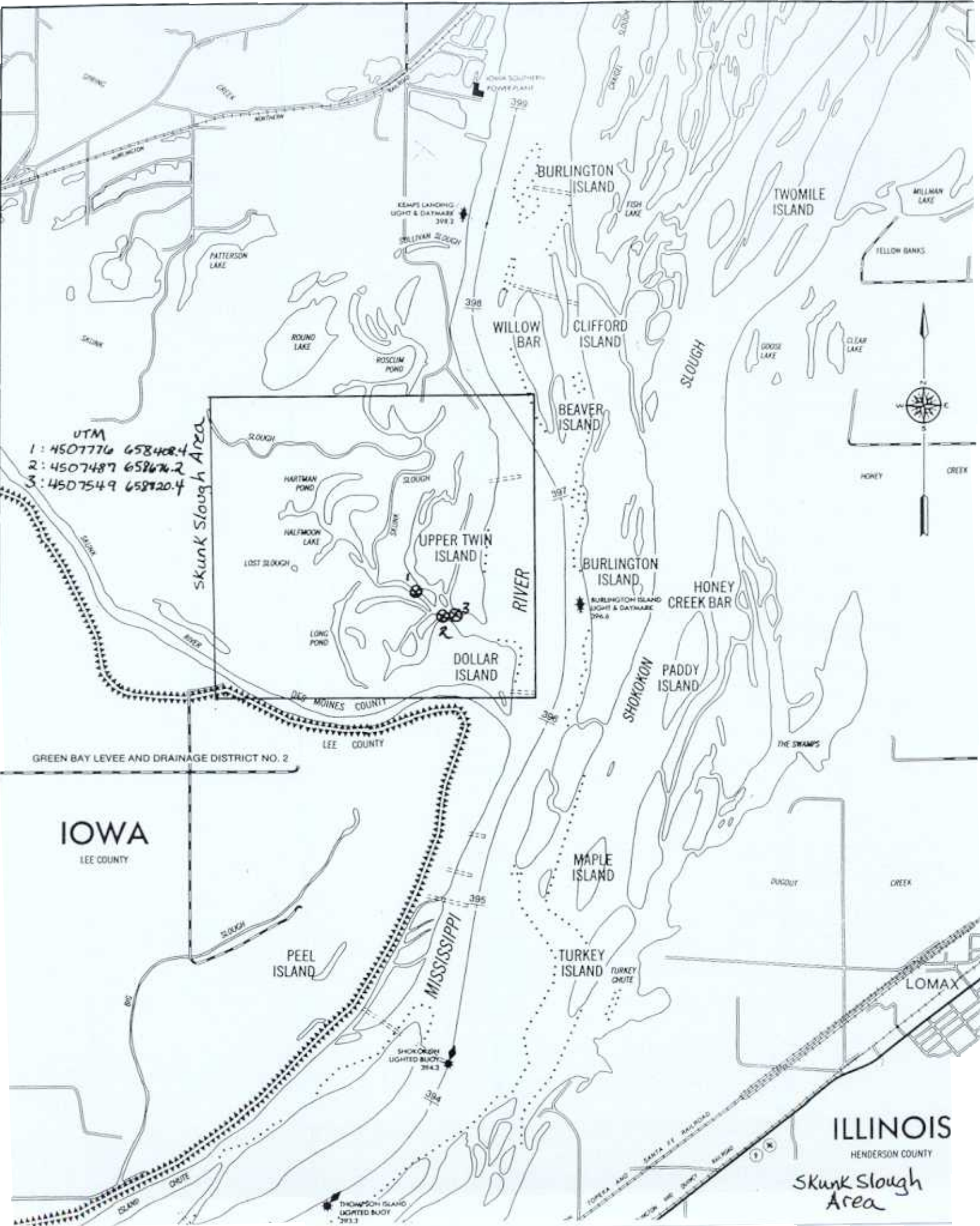
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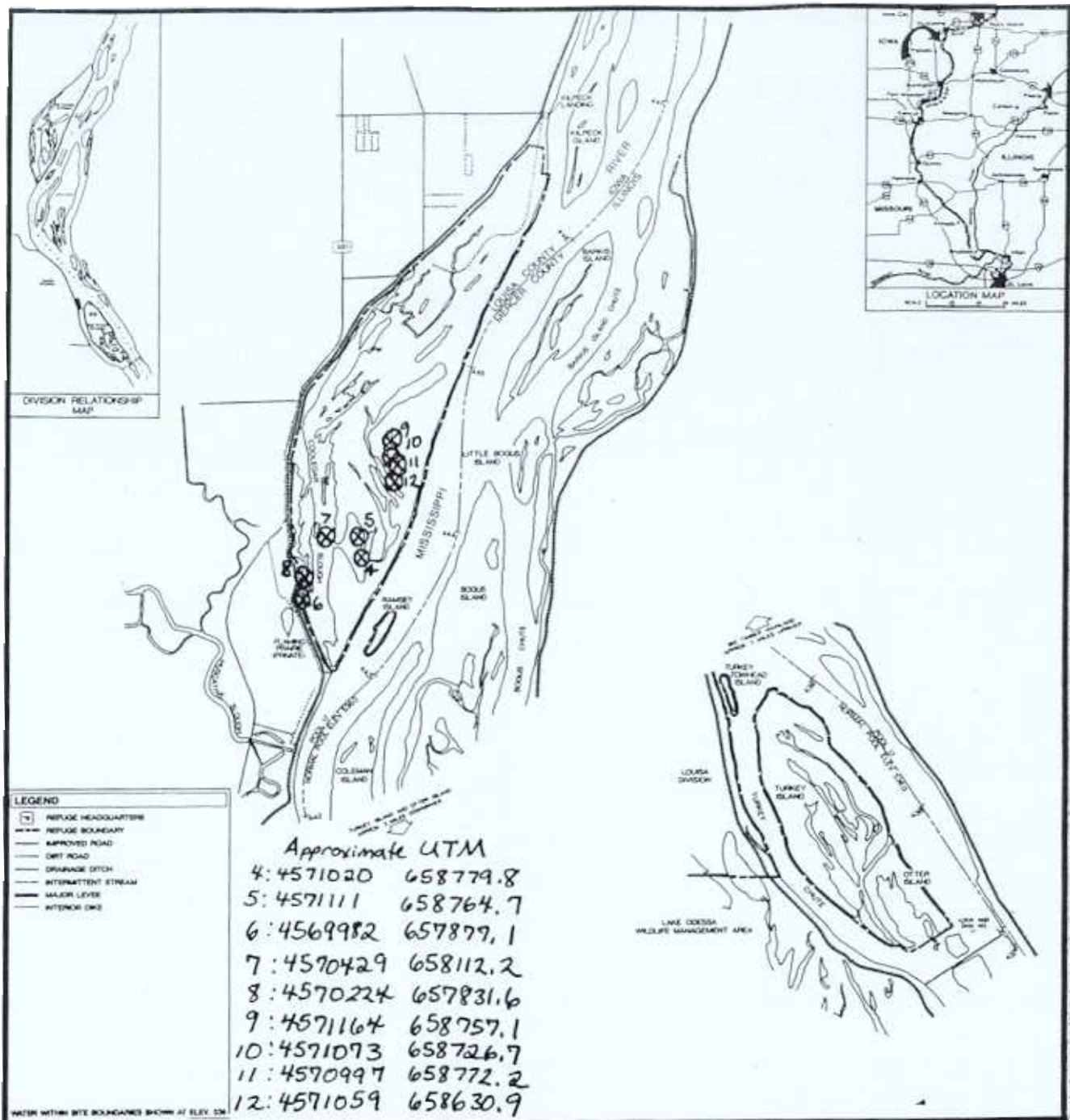
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Appendix A

Study Site Maps





BIG TIMBER DIVISION

MARK TWAIN NATIONAL WILDLIFE REFUGE

U.S. FISH AND WILDLIFE SERVICE
DEPARTMENT OF THE INTERIOR

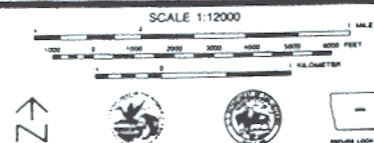


FIGURE 1

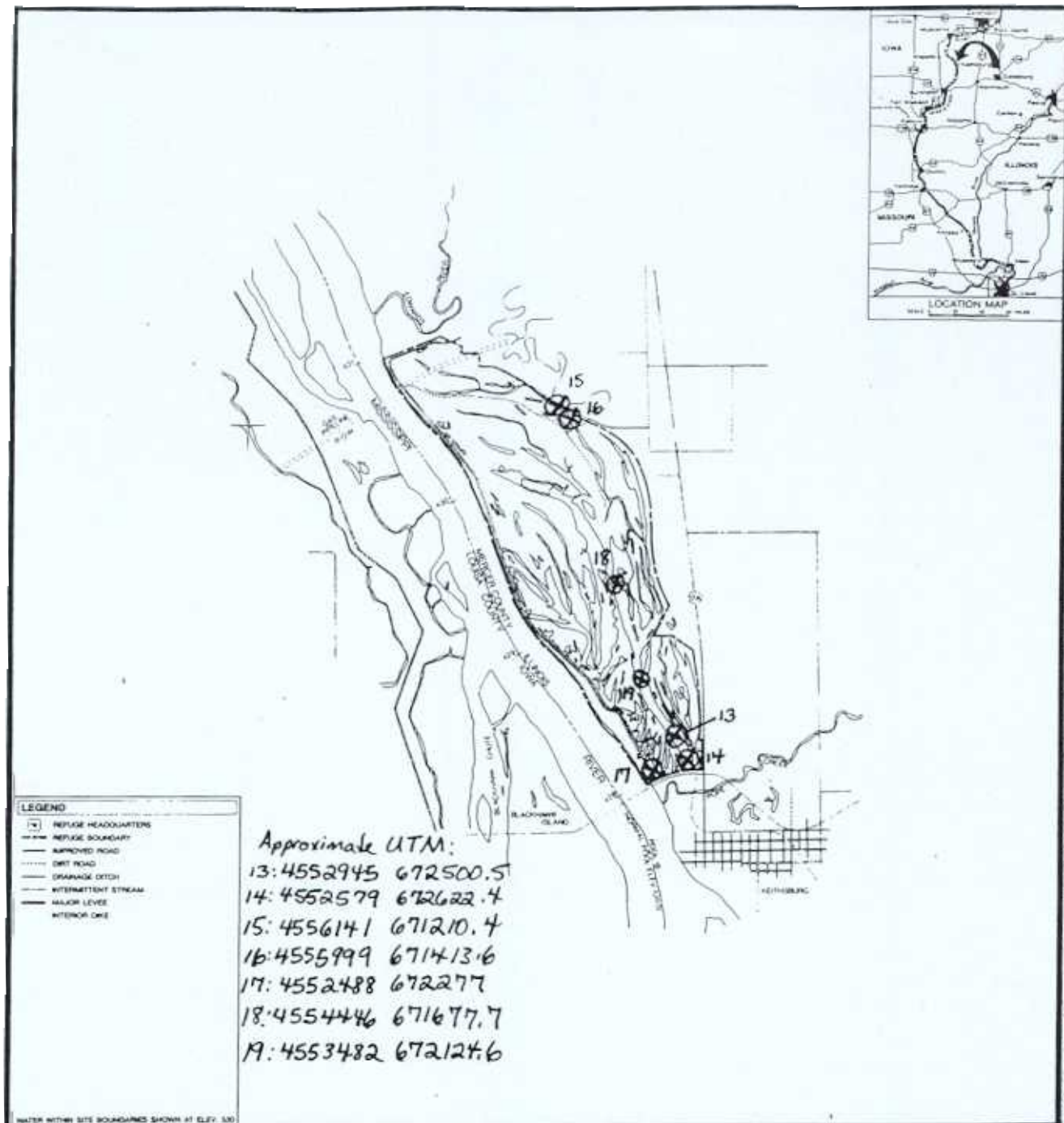
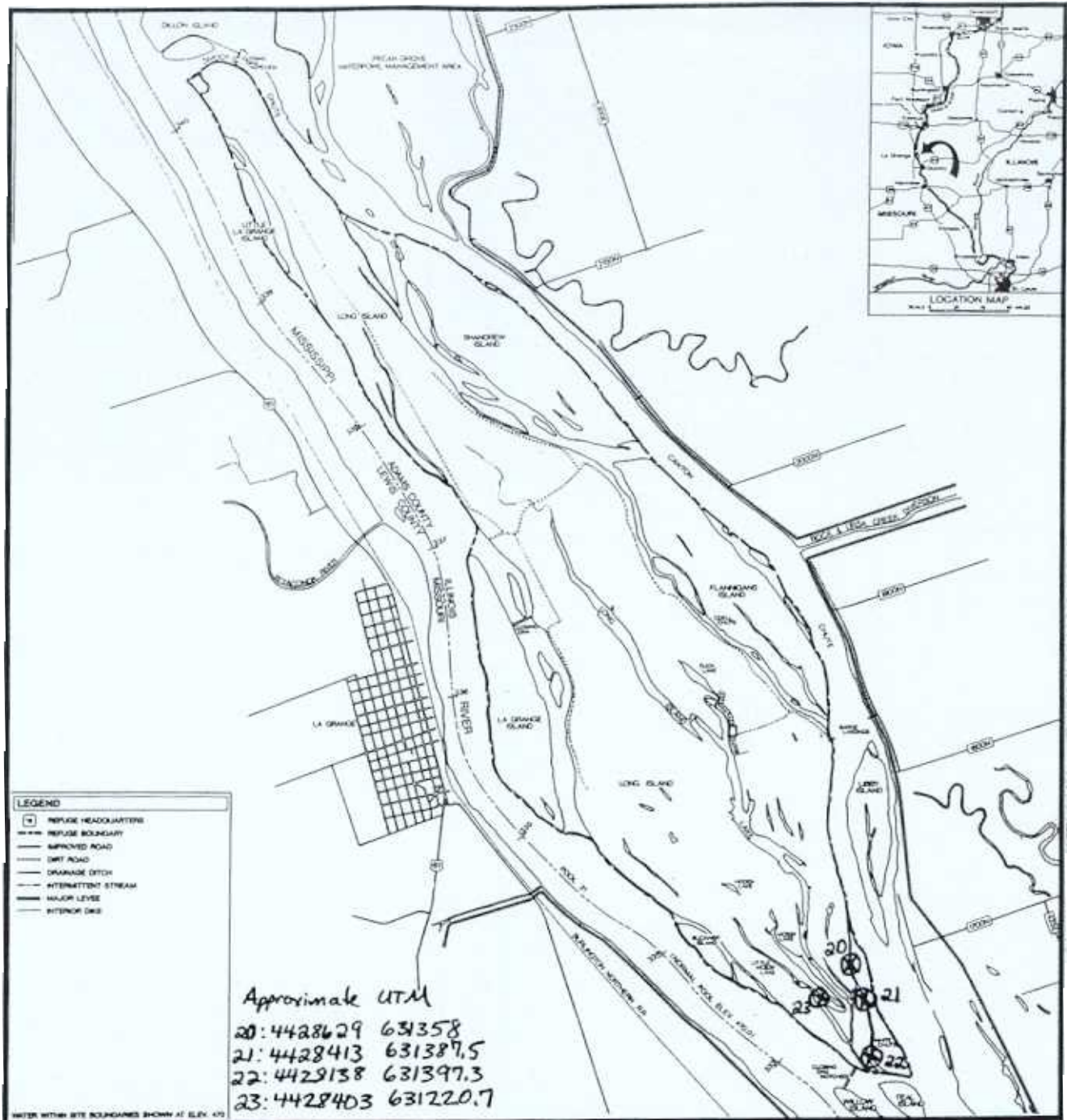
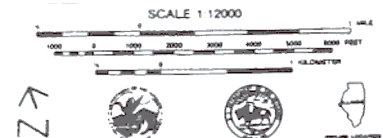


FIGURE 21



GARDNER DIVISION
 MARK TWAIN NATIONAL WILDLIFE REFUGE
 U.S. Fish and Wildlife Service
 DEPARTMENT OF THE INTERIOR



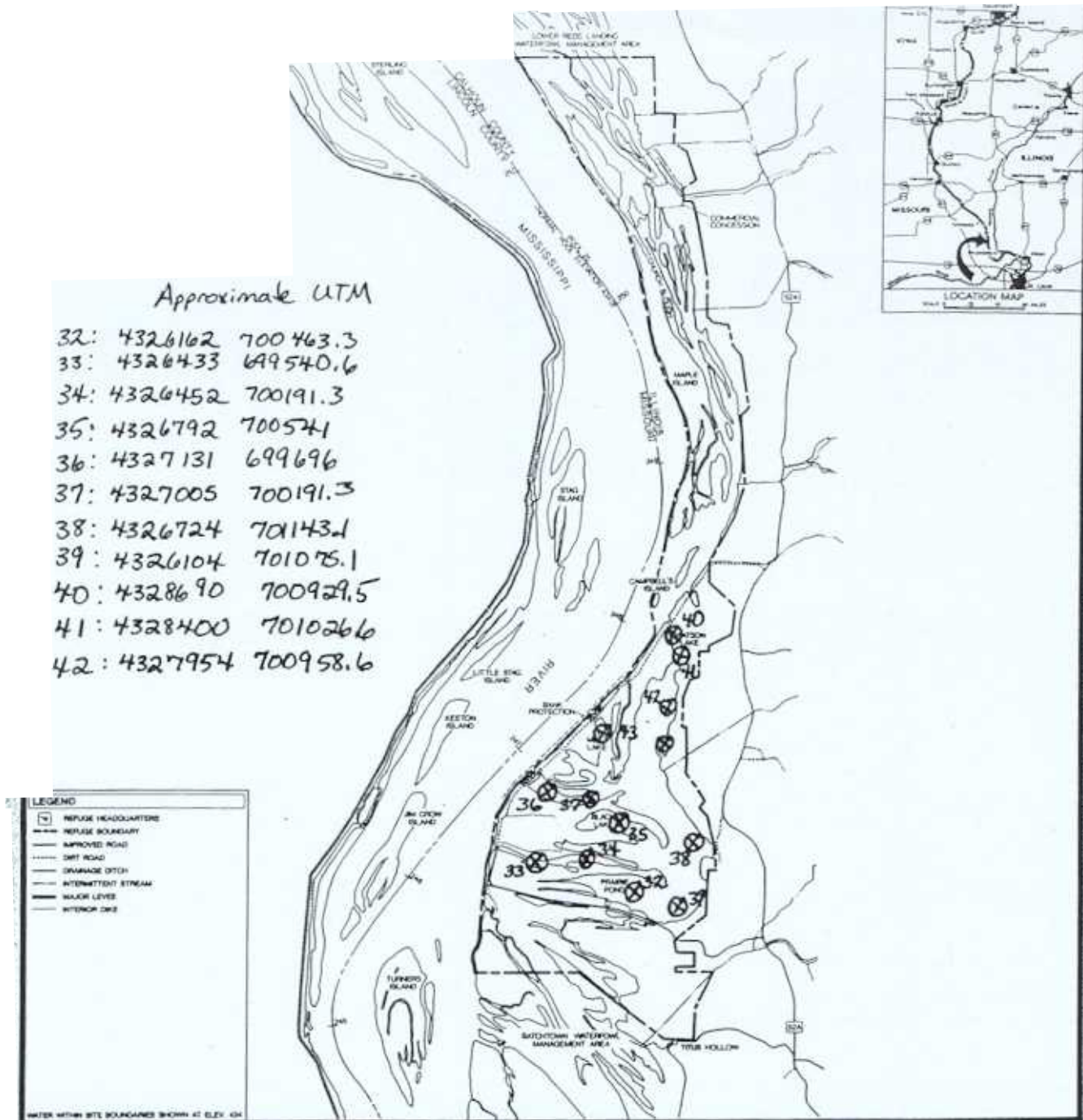


FIGURE 61

Appendix B

Contaminant and Texture Data Records

Appendix B. Contaminant concentrations (micrograms per gram, dry weight) and texture data for the sampling locations at Mark Twain National Wildlife Refuge, 1992.

| SITE | Reference No. | Zn | Pb | Mn | Cu | Cr | Cd | As | TOC | PO4 | NH3-N | % CLAY | % SILT | % SAND |
|------------------------|---------------|--------|-------|-------|-------|-------|------|------|------|---------|--------|--------|--------|--------|
| Stunt Slough | 1a | 88.80 | 51.82 | 20.90 | 26.06 | 18.91 | 0.38 | 5.84 | 2.13 | 1100.00 | 55.00 | 50.62 | 44.54 | 4.84 |
| | 2a | 116.90 | 30.48 | 26.20 | 34.65 | 21.84 | 0.48 | 7.48 | 2.73 | | 70.00 | 47.59 | 42.95 | 9.46 |
| | 3a | 84.80 | 20.79 | 19.80 | 25.88 | 14.71 | 0.41 | 6.38 | 2.03 | | 80.00 | 40.01 | 56.43 | 3.56 |
| | 1b | 95.20 | 25.12 | 21.10 | 30.38 | 16.84 | 0.43 | 6.55 | 2.86 | | | 50.80 | 44.83 | 4.37 |
| | 2b | 92.30 | 32.80 | 21.70 | 31.48 | 16.30 | 0.45 | 7.38 | 2.78 | | | 49.57 | 45.51 | 4.92 |
| Big Timber Division | 3b | 78.80 | 20.74 | 18.00 | 25.20 | 14.13 | 0.40 | 6.00 | 2.08 | | | 36.51 | 60.18 | 3.31 |
| | 4a | 86.80 | 23.44 | 19.30 | 17.56 | 13.28 | 0.37 | 4.18 | 2.18 | | 170.00 | 37.38 | 56.02 | 3.80 |
| | 5a | 107.90 | 25.43 | 25.40 | 21.80 | 17.24 | 0.48 | 5.40 | 2.38 | 780.00 | 80.00 | 38.01 | 59.09 | 2.90 |
| | 6a | 33.20 | 46.48 | 17.40 | 6.31 | 6.75 | 0.00 | 3.06 | 0.17 | | | 7.39 | 4.80 | 88.01 |
| | 7a | 108.20 | 35.32 | 26.10 | 22.40 | 22.58 | 0.45 | 5.87 | 1.79 | | 80.00 | 24.51 | 42.88 | 32.51 |
| | 8a | 88.80 | 15.82 | 16.20 | 12.34 | 9.40 | 0.27 | 4.48 | 1.74 | 580.00 | | 17.46 | 38.16 | 44.38 |
| | 9a | 127.40 | 35.38 | 30.80 | 24.64 | 26.40 | 0.46 | 6.73 | 1.53 | | | 35.88 | 58.15 | 6.18 |
| | 10a | 112.70 | 23.46 | 24.80 | 23.38 | 18.48 | 0.46 | 6.98 | 2.17 | 1000.00 | 58.00 | 32.82 | 55.67 | 11.71 |
| | 11a | 116.70 | 31.07 | 26.10 | 20.74 | 21.34 | 0.46 | 5.73 | 1.88 | 880.00 | 140.00 | 36.86 | 53.86 | 9.48 |
| | 12a | 124.80 | 30.28 | 26.80 | 24.24 | 23.96 | 0.51 | 6.33 | 1.81 | | 120.00 | 42.84 | 52.44 | 6.02 |
| | 4b | 94.80 | 27.82 | 21.20 | 18.47 | 16.08 | 0.38 | 4.40 | 2.18 | | | 36.52 | 58.46 | 5.02 |
| | 5b | 140.20 | 27.78 | 34.50 | 27.83 | 28.75 | 0.34 | 5.73 | 1.43 | | | 37.14 | 58.96 | 2.91 |
| | 6b | 31.20 | 0.00 | 13.80 | 5.92 | 7.83 | 0.00 | 3.04 | 0.48 | | | 5.83 | 3.80 | 80.37 |
| | 7b | 98.10 | 20.55 | 24.80 | 20.81 | 50.52 | 0.44 | 0.60 | 1.84 | | | 22.91 | 42.82 | 34.27 |
| | 8b | 64.40 | 17.11 | 18.80 | 14.88 | 11.02 | 0.29 | 4.29 | 2.05 | | | 20.37 | 39.39 | 40.24 |
| Keithsburg Division | 9b | 132.50 | 29.17 | 31.40 | 25.50 | 26.00 | 0.47 | 6.88 | 2.28 | | | 30.61 | 60.75 | 8.64 |
| | 10b | 124.80 | 33.15 | 28.70 | 23.10 | 22.38 | 0.47 | 5.15 | 2.30 | | | 41.86 | 45.46 | 12.88 |
| | 11b | 109.00 | 23.46 | 23.20 | 21.21 | 16.40 | 0.45 | 5.46 | 1.79 | | | 42.88 | 52.89 | 4.33 |
| | 12b | 125.40 | 24.23 | 25.90 | 25.58 | 22.57 | 0.46 | 6.13 | 1.87 | | | 40.72 | 50.48 | 8.80 |
| | 13a | 102.80 | 25.33 | 24.50 | 22.44 | 21.23 | 0.46 | 6.44 | 3.93 | | 200.00 | 28.62 | 58.06 | 13.32 |
| | 14a | 122.80 | 26.43 | 28.00 | 26.93 | 21.57 | 0.86 | 6.08 | 3.01 | | 210.00 | 38.87 | 47.83 | 13.80 |
| | 15a | 78.20 | 17.86 | 20.80 | 20.54 | 24.27 | 0.58 | 6.84 | 3.78 | 2100.00 | 290.00 | 36.52 | 57.33 | 7.15 |
| | 16a | 79.70 | 19.60 | 16.80 | 22.23 | 17.53 | 0.64 | 7.30 | 3.82 | | 320.00 | 45.32 | 48.30 | 6.38 |
| | 17a | 86.00 | 18.08 | 19.70 | 20.18 | 20.02 | 0.57 | 3.96 | 2.88 | 1900.00 | 190.00 | 31.85 | 61.96 | 6.20 |
| | 18a | 74.80 | 23.31 | 15.70 | 22.86 | 12.90 | 0.58 | 6.22 | 3.83 | | 210.00 | 38.78 | 49.78 | 10.44 |
| | 19a | 67.40 | 20.46 | 16.10 | 20.70 | 13.36 | 0.50 | 5.53 | 3.93 | | 200.00 | 32.85 | 50.95 | 16.40 |
| | 13b | 99.80 | 26.02 | 22.80 | 21.63 | 19.28 | 0.46 | 5.75 | 2.83 | | | 26.42 | 64.21 | 9.55 |
| | 14b | 128.10 | 25.81 | 35.40 | 26.18 | 38.28 | 0.76 | 6.79 | 3.67 | | | 37.30 | 49.42 | 13.28 |
| | 15b | 73.80 | 19.28 | 16.10 | 21.81 | 15.74 | 0.61 | 6.88 | 4.31 | | | 37.30 | 55.28 | 7.42 |
| | 16b | 103.80 | 21.30 | 26.30 | 26.00 | 31.32 | 0.67 | 7.33 | 4.00 | | | 55.86 | 38.29 | 6.06 |
| Gardner Division | 17b | 98.80 | 20.60 | 21.80 | 23.68 | 22.30 | 0.58 | 5.35 | 2.38 | | | 28.03 | 63.06 | 8.91 |
| | 18b | 78.70 | 22.38 | 17.00 | 23.72 | 14.82 | 0.57 | 5.83 | 3.00 | | | 36.06 | 51.26 | 8.88 |
| | 19b | 80.40 | 16.32 | 16.30 | 16.74 | 16.88 | 0.43 | 5.57 | 4.08 | | | 28.82 | 38.29 | 32.79 |
| | 20a | 18.00 | 9.26 | 0.00 | 10.34 | 3.88 | 0.30 | 2.83 | 1.58 | | | 12.17 | 57.28 | 30.55 |
| | 21a | 34.50 | 9.38 | 7.30 | 9.25 | 8.38 | 0.30 | 3.28 | 1.50 | | | 13.01 | 56.70 | 30.29 |
| | 22a | 60.70 | 11.29 | 10.70 | 11.54 | 10.08 | 0.37 | 3.82 | 1.55 | | | 16.81 | 71.80 | 11.59 |
| | 23a | 65.70 | 16.70 | 16.70 | 16.10 | 14.28 | 0.88 | 8.18 | 1.32 | 1100.00 | | 33.38 | 50.01 | 16.61 |
| | 20b | 30.80 | 7.82 | 6.80 | 7.78 | 7.97 | 0.28 | 2.78 | 1.23 | | | 8.88 | 49.74 | 40.58 |
| | 21b | 55.00 | 10.10 | 12.10 | 11.88 | 11.82 | 0.32 | 3.81 | 1.03 | | | 14.89 | 58.89 | 25.42 |
| | 22b | 56.40 | 13.15 | 11.30 | 12.77 | 10.64 | 0.41 | 4.24 | 1.07 | | | 19.19 | 72.74 | 8.07 |
| Clarence Cannon Refuge | 23b | 58.30 | 14.34 | 14.30 | 11.80 | 17.80 | 0.80 | 5.78 | 1.62 | | | 22.58 | 55.44 | 21.98 |
| | 24a | 63.50 | 23.53 | 25.80 | 21.43 | 23.26 | 0.48 | 3.34 | 2.82 | 800.00 | | 57.13 | 36.67 | 7.20 |
| | 25a | 87.00 | 17.48 | 16.80 | 21.31 | 8.54 | 0.40 | 2.58 | 3.86 | | 190.00 | 55.82 | 36.38 | 7.80 |
| | 26a | 55.30 | 17.03 | 13.00 | 21.88 | 7.91 | 0.35 | 3.06 | 2.86 | | | 58.94 | 33.46 | 7.80 |
| | 27a | 58.00 | 13.07 | 17.40 | 17.22 | 19.01 | 0.00 | 4.88 | 0.67 | | 79.00 | 45.83 | 50.16 | 4.01 |
| | 28a | 29.70 | 14.83 | 7.80 | 12.03 | 8.16 | 0.00 | 3.97 | 1.37 | | 83.00 | 23.10 | 66.81 | 11.08 |
| | 29a | 44.40 | 12.46 | 15.70 | 17.51 | 8.86 | 0.00 | 3.65 | 1.31 | | | 23.38 | 66.08 | 7.53 |
| | 30a | 89.80 | 19.48 | 18.30 | 21.84 | 18.38 | 0.88 | 3.87 | 3.35 | | 120.00 | 52.88 | 43.18 | 4.14 |
| | 31a | 78.00 | 20.31 | 20.80 | 22.77 | 12.33 | 0.86 | 4.44 | 2.80 | 980.00 | 130.00 | 57.64 | 36.26 | 6.10 |
| | 24b | 97.80 | 27.88 | 24.80 | 26.86 | 20.08 | 0.81 | 3.34 | 3.71 | | | 56.16 | 38.98 | 7.85 |
| | 25b | 83.80 | 19.83 | 18.80 | 23.53 | 12.73 | 0.44 | 2.87 | 3.11 | | | 56.80 | 38.98 | 8.52 |
| | 26b | 55.70 | 16.18 | 9.50 | 22.77 | 8.20 | 0.37 | 3.04 | 2.17 | | | 59.16 | 35.01 | 5.84 |
| | 27b | 64.80 | 15.75 | 22.00 | 16.58 | 17.58 | 0.00 | 4.88 | 1.38 | | | 35.19 | 60.39 | 4.42 |
| | 28b | 20.10 | 12.94 | 7.30 | 12.14 | 7.74 | 0.00 | 4.88 | 1.86 | | | 22.88 | 63.54 | 13.78 |
| | 30b | 77.80 | 17.93 | 20.70 | 21.91 | 11.28 | 0.86 | 3.88 | 3.30 | | | 50.83 | 43.62 | 5.56 |
| Beechtown Division | 31b | 75.40 | 17.25 | 19.70 | 21.48 | 10.77 | 0.77 | 3.88 | 3.48 | | | 55.82 | 38.21 | 6.17 |
| | 32a | 83.00 | 25.67 | 19.80 | 21.42 | 23.93 | 0.58 | 6.08 | 2.38 | | | 52.28 | 38.08 | 8.63 |
| | 33a | 80.40 | 23.15 | 22.00 | 24.58 | 14.79 | 0.57 | 6.08 | 2.41 | 790.00 | 81.00 | 45.67 | 49.78 | 4.55 |
| | 34a | 103.80 | 20.74 | 29.00 | 24.84 | 28.18 | 0.52 | 5.84 | 2.25 | | | 44.86 | 50.02 | 5.33 |
| | 35a | 145.80 | 25.26 | 37.40 | 31.17 | 35.67 | 0.81 | 7.27 | 1.86 | | 110.00 | 52.33 | 42.29 | 5.38 |
| | 36a | 106.20 | 23.23 | 29.50 | 29.08 | 22.80 | 0.55 | 7.16 | 2.47 | 1100.00 | | 50.11 | 45.33 | 4.56 |
| | 37a | 99.30 | 20.99 | 44.30 | 25.82 | 45.00 | 0.51 | 6.08 | 2.46 | 940.00 | 84.00 | 43.47 | 52.56 | 3.87 |
| | 38a | 115.00 | 26.46 | 33.50 | 27.01 | 27.48 | 0.50 | 7.20 | 1.80 | | | 48.16 | 49.08 | 2.78 |
| | 39a | 80.10 | 18.36 | 16.80 | 17.18 | 9.73 | 0.45 | 4.11 | 2.22 | 730.00 | 58.00 | 31.57 | 60.77 | 7.86 |
| | 40a | 47.80 | 11.57 | 14.70 | 10.42 | 10.73 | 0.23 | 2.78 | 1.28 | 620.00 | | 13.82 | 53.86 | 32.33 |
| | 41a | 33.00 | 10.50 | 13.10 | 8.40 | 10.04 | 0.20 | 3.41 | 0.98 | 900.00 | 40.00 | 14.97 | 81.96 | 3.07 |
| | 42a | 52.10 | 20.82 | 23.50 | 22.12 | 20.51 | 0.48 | 6.84 | 1.80 | | | 33.35 | 63.24 | 3.41 |
| | 43a | 58.80 | 17.25 | 16.40 | 18.17 | 11.00 | 0.43 | 6.34 | 2.19 | | 130.00 | 38.88 | 58.75 | 5.57 |
| | 32b | 111.10 | 26.43 | 31.70 | 26.85 | 25.87 | 0.82 | 7.44 | 2.24 | | | 47.82 | 28.27 | 15.81 |
| | 33b | 100.80 | 22.54 | 28.10 | 25.46 | 24.75 | 0.58 | 5.51 | 2.40 | | | 45.32 | 48.33 | 8.32 |
| | 34b | 126.30 | 23.05 | 30.80 | 29.80 | 26.37 | 0.52 | 5.41 | 2.38 | | | 46.86 | 50.84 | 2.80 |
| | 35b | 132.30 | 25.54 | 36.40 | 30.98 | 33.51 | 0.82 | 7.36 | 2.67 | | | 54.07 | 42.22 | 3.71 |
| | 36b | 86.30 | 21.86 | 24.00 | 23.12 | 15.80 | 0.53 | 6.42 | 2.47 | | | 50.91 | 47.27 | 1.82 |
| | 37b | 94.80 | 21.22 | 27.50 | 24.70 | 22.54 | 0.58 | 5.58 | 1.88 | | | 44.74 | 53.11 | 2.15 |
| | 38b | 98.80 | 26.38 | 29.80 | 24.84 | 21.82 | 0.49 | 7.42 | 1.88 | | | 49.80 | 48.48 | 1.62 |
| | 39b | 71.10 | 19.10 | 42.40 | 19.75 | 46.15 | 0.43 | 4.34 | 1.84 | | | 31.30 | 61.25 | 7.45 |
| | 40b | 38.40 | 10.71 | 12.00 | 9.26 | 7.31 | 0.21 | 2.38 | 0.84 | | | 14.24 | 49.88 | 36.17 |
| | 41b | 35.00 | 11.06 | 12.50 | 8.63 | 9.97 | 0.21 | 4.14 | 0.88 | | | 16.54 | 81.87 | 1.59 |
| | 42b | 61.70 | 19.88 | 18.00 | 19.48 | 12.51 | 0.44 | 6.08 | 1.51 | | | 32.80 | 63.23 | 3.87 |
| | 43b | 69.70 | 18.04 | 19.20 | 19.33 | 13.70 | 0.44 | 6.98 | 1.78 | | | 36.19 | 58.30 | 6.51 |

Appendix C
Macroinvertebrate Data Sheets

Macroinvertebrate Data Sheet

| | | | | |
|--------------------------------------|--------------------|--------------------|--------------------|--------------------|
| Reference number: Date processed: | 0-1-1-5 9-25-92 | 0-1-2-6 9-25-92 | 0-1-1-6 9-29-92 | 0-1-3-6 9-29-92 |
| Gastropods >1cm | R | R | | |
| Gastropods <1cm | A | A | A | R |
| Sphaeriidae | | 3 articulated | 4 articulated | 6 articulated |
| Hexagenia sp. | 5 | 5 | 4 | |
| Chironomidae <1cm | | | | 2 |
| Chironomidae 1-4cm | | | | 3 |
| Chironomidae >4cm | | | 1 | 3 |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |
| Reference number: Date processed: | 4-1-3-5 9-25-92 | 4-1-2-7 9-29-92 | | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | 12 | | |
| Sphaeriidae | | 1 articulated | | |
| Hexagenia sp. | 1 | 5 | | |
| Chironomidae <1cm | 16 | | | |
| Chironomidae 1-4cm | 1 | | | |
| Chironomidae >4cm | | 1 | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------------------------|--------------------|--------------------|--------------------|--|
| Reference number: Date processed: | 2-1-1- | 2-1-1- | 2-1-1- | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |
| Reference number: Date processed: | 2-1-2-7 9-29-92 | 2-1-2-6 11-7-92 | 2-1-2-8 11-7-92 | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | A | R | A | |
| Sphaeriidae | | | | |
| Hexagenia sp. | 1 | | | |
| Chironomidae <1cm | 10 | | | |
| Chironomidae 1-4cm | | 3 | 1 | |
| Chironomidae >4cm | | | | |
| Heleidae | 3 | 2 | | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

2-1-2-6 larger sp of Heleidae present

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------|----------|---------|---------|--|
| Reference number: | 2-1-4-7 | 2-1-4-6 | 2-1-4-8 | |
| Date processed: | 10-30-92 | 11-7-92 | 11-7-92 | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | 1 | | |
| Chironomidae >4cm | 3 | 3 | 1 | |
| Heleidae | | | 2 | |
| leech | | 1 | | |
| shot, Pb | | 1 | | |
| | | | | |
| Reference number: | | | | |
| Date processed: | | | | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------------------------|--------------------|---------------------|--------|--|
| Reference number: Date processed: | 2-2-1- | 2-2-1- | 2-2-1- | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |
| Reference number: Date processed: | 2-2-2-7 10-6-92 | 2-2-2-6 10-30-92 | 2-2-2- | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | 1 | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | 1 | 6 | | |
| Chironomidae >4cm | 3 | | | |
| Heleidae | 3 | 1 | | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------------------------|---------------------|--------|---------|--|
| Reference number: Date processed: | 2-2-3-6 10-19-92 | 2-2-3- | 2-2-3-8 | |
| Gastropods >1cm | R | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| <u>Hexagenia</u> sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | 3 | |
| Chironomidae >4cm | R | | | |
| Heleidae | 1 | | 4 | |
| | | | | |
| | | | | |
| | | | | |
| Reference number: Date processed: | 2-2-4- | 2-2-4- | 2-2-4- | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| <u>Hexagenia</u> sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------|----------|----------|--------|--|
| Reference number: | 3-1-1-6 | 3-1-1-4 | 3-1-1- | |
| Date processed: | 11-30-92 | 11-30-92 | | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | A | A | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| Worms | 3 | 2 | | |
| | | | | |
| | | | | |
| Reference number: | 3-1-2- | 3-1-2- | 3-1-2- | |
| Date processed: | | | | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

3-1-1-6 silk 'casts' - several

Rare= 3 or less; Common= 4 to 9 Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------------------------|---------------------|---------------------|--------|--|
| Reference number: Date processed: | 3-2-1-4 11-30-92 | 3-2-1- | 3-2-1 | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | A | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| Worms | 4 | | | |
| | | | | |
| | | | | |
| Reference number: Date processed: | 3-2-2-5 9-25-92 | 3-2-2-6 11-30-92 | 3-2-2- | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | A | A | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | 1 | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------|---------|--------|--------|--|
| Reference number: | 5-1-2-4 | 5-1-2- | 5-1-2- | |
| Date processed: | 10-6-92 | | | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | A | | | |
| Sphaeriidae | 1 | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | 1 | | | |
| Chironomidae 1-4cm | 4 | | | |
| Chironomidae >4cm | 5 | | | |
| Heleidae | 8 | | | |
| Leech | 6 | | | |
| | | | | |
| | | | | |
| Reference number: | 5-1-4- | 5-1-4- | 5-1-4- | |
| Date processed: | | | | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------|-------------------------|---------|--------|--|
| Reference number: | 5-1-6- | 5-1-6- | 5-1-6- | |
| Date processed: | | | | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| Reference number: | 5-1-8-5 | 5-1-8-4 | 5-1-8- | |
| Date processed: | 9-25-92 | 11-7-92 | | |
| Gastropods >1cm | R | R | | |
| Gastropods <1cm | C | C | | |
| Sphaeriidae | Disarticulated valves R | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | 15 | 1 | | |
| Heleidae | | 1 | | |
| Amphipod | 1 | | | |
| Leech | | 1 | | |
| | | | | |

Additional info, cite reference number:

5-1-8-4 larger 1-2 cm sp of Heleidae present

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------|---------|---------|---------|--|
| Reference number: | 5-1-10- | 5-1-10- | 5-1-10- | |
| Date processed: | | | | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |
| Reference number: | 5-1-12- | 5-1-12- | 5-1-12- | |
| Date processed: | | | | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Macroinvertebrate Data Sheet

| | | | | |
|--------------------------------------|--------|-------|--------------------|--|
| Reference number: Date processed: | 6-2-1- | 6-2-1 | 6-2-1 | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | | |
| | | | | |
| | | | | |
| | | | | |
| Reference number: Date processed: | 6-2-2 | 6-2-2 | 6-2-2-6 11-7-92 | |
| Gastropods >1cm | | | | |
| Gastropods <1cm | | | | |
| Sphaeriidae | | | | |
| Hexagenia sp. | | | | |
| Chironomidae <1cm | | | | |
| Chironomidae 1-4cm | | | | |
| Chironomidae >4cm | | | | |
| Heleidae | | | 1 | |
| | | | | |
| | | | | |
| | | | | |

Additional info, cite reference number:

Rare= 3 or less; Common= 4 to 9; Abundant= 10 to 50; Dominant= greater than 50.

Appendix D
Microtox® test Reports

FILE NAME: 3BWINTER.SPT

TEST DATE:

3-1-1-5D
Sample Description:

TEST TIME:

Sample Description:

Room temp. 22.3°C

00pm, 2-10-93, Keithsburg (Winter), Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 93.00 | 48.186 | 0.0466* |
| 2 | 89.00 | 96.371 | 0.0936 |
| 3 | 73.00 | 192.743 | 0.3333 |
| 4 | 61.00 | 385.485 | 0.5956 |
| 5 | 50.00 | 770.970 | 0.9667* |
| 6 | 31.00 | 1541.941 | 2.1398* |
| 7 | 14.00 | 3083.881 | 5.9524* |
| 8 | 5.00 | 6167.763 | 18.4667* |
| 9 | 3.00 | 12335.530 | 31.4444 |
| 10 | 3.00 | 24671.051 | 31.4444 |
| 11 | 1.00 | 49342.102 | 96.3333 |
| 12 | 1.00 | 98684.203 | 96.3333 |

| | | | |
|----------------|-------|--------|-------|
| CONTROL It's : | 96.00 | 101.00 | 95.00 |
|----------------|-------|--------|-------|

[illegible]

*SLOPE = 1.4334

ANN

1 > 5

Figure 1 is a dose-response graph showing the inhibition of ³H-thymidine incorporation by 100% ethanol. The x-axis represents 'CONCENTRATION' on a logarithmic scale, with major ticks at 100, 1000, 10E4, and 10E5. The y-axis represents relative activity, with '10' at the top and '1' at the bottom. Two sigmoidal curves are plotted: the upper curve is labeled 'EC50' and the lower curve is labeled '100% ethanol'. The EC50 curve is shifted to the right of the 100% ethanol curve, indicating a higher concentration is required for 50% inhibition.

EC50 853.555 (95% CONFIDENCE RANGE 654.224 TO 1113.617)

Used for calculations

* Invalid games

FILE NAME: 2-1-3-3A.SPT

TEST TIME: _____

12:55pm, 1-29-93, Round Pond, Detailed Test, Dupe A

Osmotic Adjustment: none

Dilution Factor : 2

Concentration Units: ppm

| NUMBER | 1c | CONC. | GAMMA |
|--------|--------|-----------|----------|
| 1 | 98.00 | 48.186 | 0.0034* |
| 2 | 104.00 | 96.371 | -0.0545* |
| 3 | 95.00 | 192.743 | 0.0351* |
| 4 | 93.00 | 385.485 | 0.0573 |
| 5 | 98.00 | 770.970 | 0.0034* |
| 6 | 91.00 | 1541.941 | 0.0806 |
| 7 | 91.00 | 3083.881 | 0.0806 |
| 8 | 105.00 | 6167.763 | -0.0635* |
| 9 | 66.00 | 12335.530 | 0.4899* |
| 10 | 58.00 | 24671.051 | 0.6954* |
| 11 | 35.00 | 49342.102 | 1.8095* |
| 12 | 16.00 | 98684.203 | 5.1458* |

| | | | |
|----------------|-------|-------|--------|
| CONTROL It's : | 95.00 | 96.00 | 104.00 |
|----------------|-------|-------|--------|

```

10 *****
   *SLOPE = 1.1558

```

The figure is a log-log plot showing the relationship between Gamma (Y-axis) and Concentration (X-axis). The Y-axis ranges from 10 to 1000, and the X-axis ranges from 10E4 to 10E6. The plot displays a series of horizontal lines at different Gamma levels, labeled 9, 10, 11, and 12. A point labeled 'EC50' is marked on the line at Gamma 10. The lines are horizontal, indicating that Gamma is constant for a given concentration range.

EC5027454.191 (95% CONFIDENCE RANGE:16731.852 TO 45047.773)

Used for calculations
* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: 2-1-1-3B.SPT

TEST DATE: _____

TEST TIME: _____

Sample Description:

3:15pm, 1-27-93, Round Pond, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: n

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units:

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 97.00 | 48.186 | -0.0034* |
| 2 | 95.00 | 96.371 | 0.0175* |
| 3 | 92.00 | 192.743 | 0.0507 |
| 4 | 78.00 | 385.485 | 0.2393 |
| 5 | 68.00 | 770.970 | 0.4216# |
| 6 | 45.00 | 1541.941 | 1.1481# |
| 7 | 25.00 | 3083.881 | 2.8667# |
| 8 | 10.00 | 6167.763 | 8.6667# |
| 9 | 2.00 | 12335.530 | 47.3333 |
| 10 | 1.00 | 24671.051 | 95.6667 |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

| | | | |
|----------------|-------|-------|--------|
| CONTROL It's : | 95.00 | 94.00 | 101.00 |
|----------------|-------|-------|--------|

```
10 OAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAC
   *SLOPE = 1.4405                                     8
```

EC50 1417.029 (95% CONFIDENCE RANGE: 1284.887 TO 1562.760)

* Used for calculations
* Invalid gammas

FILE NAME: 2-1-1-3A.SPT

TEST DATE:

TEST TIME: _____

Sample Description:

2:30^{PM} 1-27-93, Round Pond, Detailed Test, ^{Room Temp.} Dupe A. 21.7°C

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|-----------|
| 1 | 155.00 | 48.186 | -0.2968* |
| 2 | 116.00 | 96.371 | -0.0603* |
| 3 | 107.00 | 192.743 | 0.0187* |
| 4 | 100.00 | 385.485 | 0.0900* |
| 5 | 87.00 | 770.970 | 0.2529* |
| 6 | 72.00 | 1541.941 | 0.5139* |
| 7 | 35.00 | 3083.881 | 2.1143* |
| 8 | 15.00 | 6167.763 | 6.2667* |
| 9 | 3.00 | 12335.530 | 35.3333* |
| 10 | 1.00 | 24671.051 | 108.0000* |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

```
CONTROL It's :   95.00   135.00   97.00  
10 0#####9 #####  
*SLOPE = -1.6863
```

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. EC50

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100
1000 CONCENTRATION 10E4 10E5

EC50 1848.161 (95% CONFIDENCE RANGE: 1502.184 TO 2273.823)

Used for calculations
* Invalid gammas

FILE NAME: 0-1-2-3B.SPT

TEST TIME:

1e Description: room temp. 22.0°C
1:10, 1-15-93, pool 19, Detailed Test. Dupe

Osmotic Adjustment: none

Dilution Factor : 2

Concentration Units: ppm

| NUMBER | It | COWC. | GAMMA |
|--------|--------|-----------|----------|
| 1 | 63.00 | 48.186 | 0.4656 |
| 2 | 117.00 | 96.371 | -0.2108* |
| 3 | 59.00 | 192.743 | 0.5650* |
| 4 | 45.00 | 385.485 | 1.0519* |
| 5 | 25.00 | 770.970 | 2.6933* |
| 6 | 12.00 | 1541.941 | 6.6944* |
| 7 | 2.00 | 3083.881 | 45.1667 |
| 8 | 4.00 | 6167.763 | 22.0833 |
| 9 | 2.00 | 12335.530 | 45.1667 |
| 10 | 1.00 | 24671.051 | 91.3333 |
| 11 | 1.00 | 49342.102 | 91.3333 |
| 12 | 0.00 | 98684.203 | > 999 * |

| | | | |
|----------------|-------|-------|-------|
| CONTROL It's : | 96.00 | 96.00 | 85.00 |
|----------------|-------|-------|-------|

10 OAAA

*SLOPE - 1.2057

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[illegible]

EC50 334.646 (95% CONFIDENCE RANGE 262.136 TO 427.212)

Used for calculations

* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: 0-1-3-3B.SPT

TEST DATE:

TEST TIME: _____

Sample Description:

2:15, 1-8-93, pool 19, Detailed Test, Dupe B Room Temp. 24.6 °C

Procedure: SOLID-PHASE

Initial Concentration : 98684.2 ppm

Test Time: 5 minutes

Osmotic Adjustment: none

Dilution Factor : 2

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|----------|
| 1 | 106.00 | 48.186 | -0.0031* |
| 2 | 92.00 | 96.371 | 0.1486 |
| 3 | 89.00 | 192.743 | 0.1873# |
| 4 | 77.00 | 385.485 | 0.3723# |
| 5 | 59.00 | 770.970 | 0.7910# |
| 6 | 37.00 | 1541.941 | 1.8559# |
| 7 | 25.00 | 3083.881 | 3.2267# |
| 8 | 6.00 | 6167.763 | 16.6111 |
| 9 | 3.00 | 12335.530 | 34.2222 |
| 10 | 2.00 | 24671.051 | 51.8333 |
| 11 | 2.00 | 49342.102 | 51.8333 |
| 12 | 0.00 | 98684.203 | > 999 * |

CONTROL It's : 97.00 117.00 103.00

10 *****
"SLOPE = 1.0531

*SLOPE = 1.0531

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EC50 950.852 (95% CONFIDENCE RANGE: 854.140 TO 1058.515)

Used for calculations
* Invalid gammas

FILE NAME: 0-1-2-3A.SPT

TEST TIME: _____

12:10pm, 1-15-93, pool 19, Detailed Test, Dupe A

Repro + temp. 22.0°C

Osmotic Adjustment: none

Dilution Factor : 2

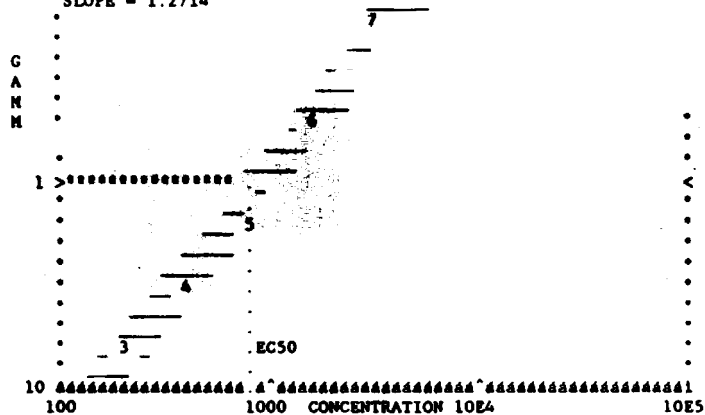
Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 87.00 | 48.186 | -0.0460* |
| 2 | 57.00 | 96.371 | 0.4561 |
| 3 | 70.00 | 192.743 | 0.1857# |
| 4 | 60.00 | 385.485 | 0.3833# |
| 5 | 48.00 | 770.970 | 0.7292# |
| 6 | 27.00 | 1541.941 | 2.0741# |
| 7 | 11.00 | 3083.881 | 6.5455# |
| 8 | 2.00 | 6167.763 | 40.5000 |
| 9 | 1.00 | 12335.530 | 82.0000 |
| 10 | 0.00 | 24671.051 | > 999 * |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

| | | | |
|----------------|-------|-------|-------|
| CONTROL It's : | 94.00 | 79.00 | 76.00 |
|----------------|-------|-------|-------|

10 OAAA

*SLOPE = 1.2714



EC50 813.897 (95% CONFIDENCE RANGE 649.133 TO 1020.483)

* Used for calculations

* Invalid gammas

FILE NAME: 2-2-1-3A.SPT

TEST TIME:

Room Temp 21.4°C

Procedure: SOLID-PHASE

Initial Concentration : 98684.2 ppm

Test Time: 5 minutes

Concentration Units: ppm

| | | | |
|----------------|-------|-------|-------|
| CONTROL It's : | 95.00 | 97.00 | 99.00 |
|----------------|-------|-------|-------|

*SLOPE = 1.5863

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. EC50

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100
1000 CONCENTRATION 10E4
10E5

EC50 2667.740 (95% CONFIDENCE RANGE: 2096.438 TO 3394.727)

```
# Used for calculations
* Invalid gammas
```

FILE NAME: 2-2-1-3B.SPT

TEST DATE:

TEST TIME:

Sample Description:

Room Temp. 21.4°C .

45pm, 1-29-93, Little Denny Pond, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

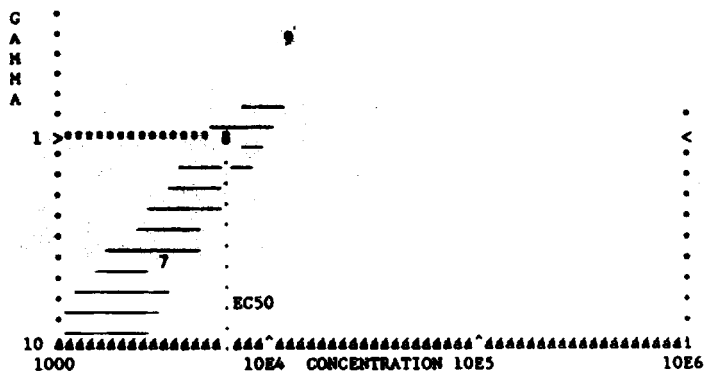
Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|-------------------|--------|--------------------|-----------|
| 1 | 131.00 | 48.186 | -0.2188* |
| 2 | 123.00 | 96.371 | -0.1680* |
| 3 | 133.00 | 192.743 | -0.2306* |
| 4 | 140.00 | 385.485 | -0.2690* |
| 5 | 118.00 | 770.970 | -0.1328* |
| 6 | 105.00 | 1541.941 | -0.0254* |
| 7 | 80.00 | 3083.881 | 0.2792* |
| 8 | 51.00 | 6167.763 | 1.0065* |
| 9 | 21.00 | 12335.530 | 3.8730* |
| 10 | 12.00 | 24671.051 | 7.5278* |
| 11 | 9.00 | 49342.102 | 10.3704* |
| 12 | 1.00 | 98684.203 | 101.3333* |
| CONTROL It's : | | 96.00 135.00 76.00 | |
| 10 ***** 11 ***** | | | |
| *SLOPE = 1.3333 | | | |



EC50 6581.972 (95% CONFIDENCE RANGE: 3927.031 TO 11031.833)

Used for calculations
* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: 2-2-4-3A.SPT

TEST DATE: _____

Sample Description:

TEST TIME: _____

Room temp. 21.4°C

2:00pm, 1-29-93, Little Denny Pond, Detailed Test, Dupe A

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 36.00 | 48.186 | 1.3611 |
| 2 | 93.00 | 96.371 | -0.0860* |
| 3 | 86.00 | 192.743 | -0.0116* |
| 4 | 72.00 | 385.485 | 0.1806* |
| 5 | 49.00 | 770.970 | 0.7347 |
| 6 | 27.00 | 1541.941 | 2.1481 |
| 7 | 7.00 | 3083.881 | 11.1429 |
| 8 | 1.00 | 6167.763 | 84.0000 |
| 9 | 1.00 | 12335.530 | 84.0000 |
| 10 | 0.00 | 24671.051 | > 999 * |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 1.00 | 98684.203 | 84.0000 |

CONTROL It's : 96.00 72.00 87.00

MICROTOX DATA REPORT

FILE NAME: 2-2-4-3A.SPT

TEST DATE: _____

Sample Description:

TEST TIME: _____

2:00pm, 1-29-93, Little Denny Pond, Detailed Test, Dupe A

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 36.00 | 48.186 | 1.3611 |
| 2 | 93.00 | 96.371 | -0.0860* |
| 3 | 86.00 | 192.743 | -0.0116* |
| 4 | 72.00 | 385.485 | 0.1806* |
| 5 | 49.00 | 770.970 | 0.7347 |
| 6 | 27.00 | 1541.941 | 2.1481 |
| 7 | 7.00 | 3083.881 | 11.1429 |
| 8 | 1.00 | 6167.763 | 84.0000 |
| 9 | 1.00 | 12335.530 | 84.0000 |
| 10 | 0.00 | 24671.051 | > 999 * |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 1.00 | 98684.203 | 84.0000 |

CONTROL It's : 96.00 72.00 87.00

MICROTOX DATA REPORT

FILE NAME: 2-2-4-3A.SPT

TEST DATE: _____

Sample Description:

TEST TIME: _____

FILE NAME: 2-2-4-3B.SPT

TEST DATE:

TEST TIME:

Sample Description:

Room temp. 21.5°C

2:45pm, 1-29-93, Little Denny Pond, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|---------|
| 1 | 92.00 | 48.186 | 0.0688 |
| 2 | 90.00 | 96.371 | 0.0926 |
| 3 | 76.00 | 192.743 | 0.2939 |
| 4 | 72.00 | 385.485 | 0.3657* |
| 5 | 44.00 | 770.970 | 1.2348* |
| 6 | 19.00 | 1541.941 | 4.1754* |
| 7 | 4.00 | 3083.881 | 23.5833 |
| 8 | 0.00 | 6167.763 | > 999 * |
| 9 | 0.00 | 12335.530 | > 999 * |
| 10 | 0.00 | 24671.051 | > 999 * |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

CONTROL It's : 96.00 101.00 98.00

```

10 #####
   *SLOPE = 1.7565

```

Figure 1 is a log-log plot showing the relationship between GAMMA (Y-axis) and CONCENTRATION (X-axis). The Y-axis ranges from 10 to 100, and the X-axis ranges from 100 to 10E5. The plot displays a sigmoidal curve with points labeled 1, 2, 3, 4, and 5. A horizontal dashed line is labeled EC50.

EC50 683.532 (95% CONFIDENCE RANGE: 683.532 TO 683.532)

```
# Used for calculations
* Invalid gammas
```

FILE NAME: 3-1-1-3A.SPT

TEST DATE:

TEST TIME:

Room temp 21.4°C

4:00pm, 1-6-93, Keithsburg-lower, Detailed Test, Dupe A

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|----------|
| 1 | 100.00 | 48.186 | -0.0567* |
| 2 | 92.00 | 96.371 | 0.0254* |
| 3 | 94.00 | 192.743 | 0.0035* |
| 4 | 79.00 | 385.485 | 0.1941 |
| 5 | 73.00 | 770.970 | 0.2922* |
| 6 | 55.00 | 1541.941 | 0.7152* |
| 7 | 32.00 | 3083.881 | 1.9479* |
| 8 | 14.00 | 6167.763 | 5.7381* |
| 9 | 9.00 | 12335.530 | 9.4815 |
| 10 | 5.00 | 24671.051 | 17.8667 |
| 11 | 2.00 | 49342.102 | 46.1667 |
| 12 | 1.00 | 98684.203 | 93.3333 |

| | | | |
|----------------|-------|-------|--------|
| CONTROL It's : | 97.00 | 82.00 | 104.00 |
|----------------|-------|-------|--------|

```

10 0#####
  *SLOPE = 1.4332
  
```

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. EC50

10
100
1000
CONCENTRATION
10E4
10E5

EC50 1881.132 (95% CONFIDENCE RANGE: 1701.806 TO 2079.355)

Used for calculations
* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: 3-1-1-3B.SPT

TEST DATE: _____

Sample Description:

TEST TIME: _____

Room temp 21.5°C

4:20pm, 1-6-93, Keithsburg-lower, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|---------|
| 1 | 103.00 | 48.186 | 0.0453* |
| 2 | 101.00 | 96.371 | 0.0660 |
| 3 | 101.00 | 192.743 | 0.0660 |
| 4 | 92.00 | 385.485 | 0.1703* |
| 5 | 75.00 | 770.970 | 0.4356* |
| 6 | 55.00 | 1541.941 | 0.9576* |
| 7 | 28.00 | 3083.881 | 2.8452* |
| 8 | 10.00 | 6167.763 | 9.7667* |
| 9 | 2.00 | 12335.530 | 52.8333 |
| 10 | 2.00 | 24671.051 | 52.8333 |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

CONTROL It's : 95.00 113.00 115.00

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*SLOPE = 1.4391

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EC50

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100 1000 CONCENTRATION 10E4 10E5

EC50 1403.855 (95% CONFIDENCE RANGE: 1206.177 TO 1633.929)

Used for calculations
* Invalid gammas

FILE NAME: 3-2-2-3A.SPT

TEST DATE:

TEST TIME:

Room Temp. 25°C

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

EC50 1522.536 (95% CONFIDENCE RANGE: 1289.541 TO 1797.630)

Used for calculations
* Invalid gammas

FILE NAME: 3-2-2-3B.SPT

TEST DATE:

TEST TIME:

Sample Description:

Room temp 24.8°C

1:30pm, 1-8-93, Keithsburg-upper, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|-----------|
| 1 | 100.00 | 48.186 | 0.0200* |
| 2 | 101.00 | 96.371 | 0.0099* |
| 3 | 95.00 | 192.743 | 0.0737 |
| 4 | 85.00 | 385.485 | 0.2000# |
| 5 | 62.00 | 770.970 | 0.6452# |
| 6 | 34.00 | 1541.941 | 2.0000# |
| 7 | 19.00 | 3083.881 | 4.3684 |
| 8 | 8.00 | 6167.763 | 11.7500 |
| 9 | 3.00 | 12335.530 | 33.0000 |
| 10 | 2.00 | 24671.051 | 50.0000 |
| 11 | 1.00 | 49342.102 | 101.0000* |
| 12 | 1.00 | 98684.203 | 101.0000* |

CONTROL It's : 96.00 109.00 101.00

SLOPE = 1.6610

EC50

1
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100 1000 10E4 10E5

CONCENTRATION

EC50 1011.776 (95% CONFIDENCE RANGE: 940.898 TO 1087.993)

* Used for calculations
* Invalid gammas

FILE NAME: 4-1-1-3A.SPT

TEST DATE: _____
TEST TIME: _____
Room Temp. 22.3°C

Sample Description: Room Temp. 22.3°C
1:30pm, 1-22-93, White Chute, Detailed Test, Dupe A

Osmotic Adjustment: none

Dilution Factor : 2

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|----------------|-------|-----------|----------|
| 1 | 86.00 | 48.186 | 0.0736 |
| 2 | 79.00 | 96.371 | 0.1688 |
| 3 | 90.00 | 192.743 | 0.0259* |
| 4 | 91.00 | 385.485 | 0.0147* |
| 5 | 39.00 | 770.970 | 1.3675* |
| 6 | 84.00 | 1541.941 | 0.0992* |
| 7 | 80.00 | 3083.881 | 0.1542* |
| 8 | 47.00 | 6167.763 | 0.9645* |
| 9 | 50.00 | 12335.530 | 0.8467* |
| 10 | 30.00 | 24671.051 | 2.0778* |
| 11 | 15.00 | 49342.102 | 5.1556* |
| 12 | 7.00 | 98684.203 | 12.1905* |
| CONTROL It's : | 95.00 | 94.00 | 88.00 |

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*SLOPE = 0.7340

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EC50 8044.636 (95% CONFIDENCE RANGE: 2914.395 TO 22205.693)

Used for calculations
* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: 4-1-1-3B.SPT

TEST DATE: _____
TEST TIME: _____

TEST TIME: _____

Sample Description:

2:15pm, 1-22-93, White Chute, Detailed Test, Dupe B Room Temp. 22.3°C

Room temp. 22.3°C
Test Dura B

Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm
Test Time: 5 minutes

Dilution Factor : 2

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------------|-------|-----------|---------|
| 1 | 94.00 | 48.186 | 0.0638 |
| 2 | 95.00 | 96.371 | 0.0526 |
| 3 | 98.00 | 192.743 | 0.0204* |
| 4 | 90.00 | 385.485 | 0.1111 |
| 5 | 90.00 | 770.970 | 0.1111 |
| 6 | 93.00 | 1541.941 | 0.0753 |
| 7 | 82.00 | 3083.881 | 0.2195 |
| 8 | 79.00 | 6167.763 | 0.2658# |
| 9 | 54.00 | 12335.530 | 0.8519# |
| 10 | 33.00 | 24671.051 | 2.0303# |
| 11 | 18.00 | 49342.102 | 4.5556# |
| 12 | 11.00 | 98684.203 | 8.0909 |
| CONTROL It's | 95.00 | 100.00 | 0.0000 |

| | | | |
|----------------|-------|--------|-------|
| CONTROL It's : | 95.00 | 108.00 | 97.00 |
|----------------|-------|--------|-------|

*SLOPE - 1.3550

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EC50

1000 10E4 10E5 10E6

CONCENTRATION

EC5015236.967 (95% CONFIDENCE RANGE:12339.940 TO 18814.123)

* Used for calculation

* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: 4-1-3-3A.SPT

TEST DATE:

TEST TIME:

Sample Description:

12:10, 1-22-93, White Chute, Detailed Test, Dupe A

Room temp. 22.1°C

Procedure: SOLID-PHASE

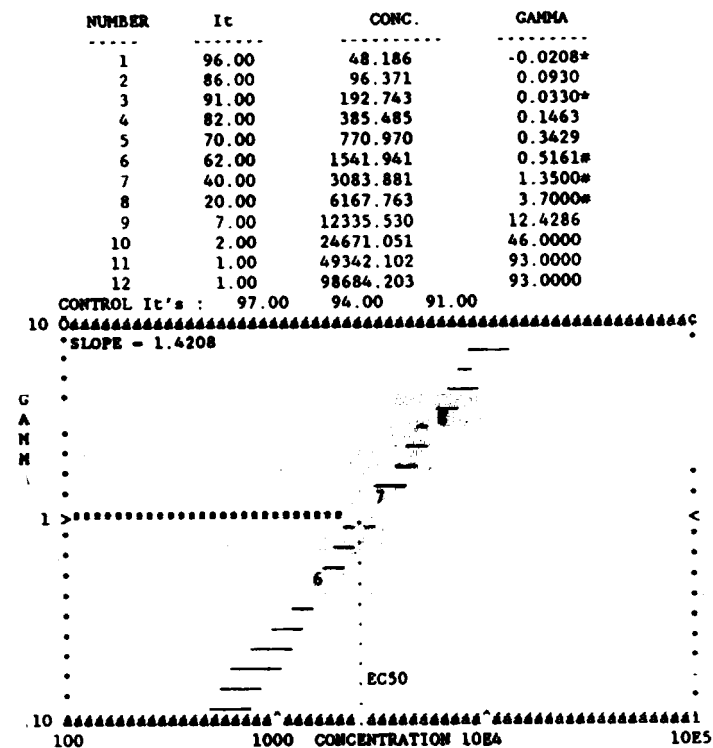
Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm



EC50 2469.598 (95% CONFIDENCE RANGE: 2208.433 TO 2761.648)

* Used for calculations

* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: 4-1-3-3B.SPT

TEST DATE:

Sample Description:

TEST TIME:

1:00, 1-22-93, White Chute, Detailed Test, Dupe B

Room temp. 22.2°C

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|----------|
| 1 | 130.00 | 48.186 | -0.1128* |
| 2 | 114.00 | 96.371 | 0.0117* |
| 3 | 116.00 | 192.743 | -0.0057* |
| 4 | 114.00 | 385.485 | 0.0117* |
| 5 | 72.00 | 770.970 | 0.6019* |
| 6 | 0.00 | 1541.941 | > 999 * |
| 7 | 70.00 | 3083.881 | 0.6476* |
| 8 | 49.00 | 6167.763 | 1.3537* |
| 9 | 22.00 | 12335.530 | 4.2424* |
| 10 | 7.00 | 24671.051 | 15.4762* |
| 11 | 4.00 | 49342.102 | 27.8333* |
| 12 | 3.00 | 98684.203 | 37.4444 |

| | | | |
|----------------|-------|--------|--------|
| CONTROL It's : | 96.00 | 126.00 | 124.00 |
|----------------|-------|--------|--------|

[illegible]

*SLOPE = 0.2535

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10
100 1000 CONCENTRATION 10E4 10E5

EC50 544.984 (95% CONFIDENCE RANGE: 12.458 TO 23840.254)

```
# Used for calculations
* Invalid gammas
```

MICROTOX DATA REPORT

FILE NAME: 5-1-1-3A.SPT

TEST DATE:

TEST TIME: _____

Sample Description:

Room temp. 23.4°C

3:30pm, 2-3-93, Batchtown I and II, Detailed Test, Dupe A

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 90.00 | 48.186 | 0.0926 |
| 2 | 79.00 | 96.371 | 0.2447 |
| 3 | 73.00 | 192.743 | 0.3470# |
| 4 | 50.00 | 385.485 | 0.9667# |
| 5 | 22.00 | 770.970 | 3.4697# |
| 6 | 6.00 | 1541.941 | 15.3889# |
| 7 | 1.00 | 3083.881 | 97.3333 |
| 8 | 1.00 | 6167.763 | 97.3333 |
| 9 | 0.00 | 12335.530 | > 999 * |
| 10 | 0.00 | 24671.051 | > 999 * |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

CONTROL It's : 96.00 98.00 101.00

SLOPE = 1.8256

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CONCENTRATION

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EC50 368.185 (95% CONFIDENCE RANGE: 296.380 TO 457.388)

```
# Used for calculations
* Invalid gammas
```

MICROTOX DATA REPORT

FILE NAME: 5-1-1-3B.SPT

TEST DATE: _____

TEST TIME: _____

Sample Description:

Room Temp: 23.4°C .

4:00pm, 2-3-93, Batchtown I and II, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|-----------|
| 1 | 96.00 | 48.186 | 0.1181 |
| 2 | 83.00 | 96.371 | 0.2932 |
| 3 | 76.00 | 192.743 | 0.4123 |
| 4 | 57.00 | 385.485 | 0.8830* |
| 5 | 31.00 | 770.970 | 2.4624* |
| 6 | 12.00 | 1541.941 | 7.9444* |
| 7 | 4.00 | 3083.881 | 25.8333* |
| 8 | 2.00 | 6167.763 | 52.6667 |
| 9 | 1.00 | 12335.530 | 106.3333* |
| 10 | 1.00 | 24671.051 | 106.3333* |
| 11 | 1.00 | 49342.102 | 106.3333* |
| 12 | 0.00 | 98684.203 | > 999 * |

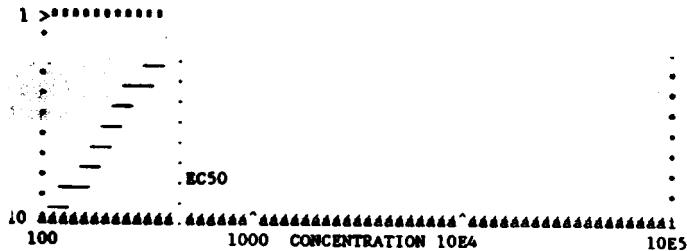
| | | | |
|----------------|-------|--------|--------|
| CONTROL It's : | 95.00 | 114.00 | 113.00 |
|----------------|-------|--------|--------|

[illegible]

*SLOPE - 1.6302

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EC50 428.209 (95% CONFIDENCE RANGE 379.531 TO 483.129)

Used for calculations

* Invalid gammas

FILE NAME: 5-1-4-3A.SPT

TEST TIME:

Room temp. 23.5°C

2:25pm, 2-3-93, Batchtown I and II, Detailed Test, Dupe A

Osmotic Adjustment: none

Dilution Factor : 2

Concentration Units: ppm

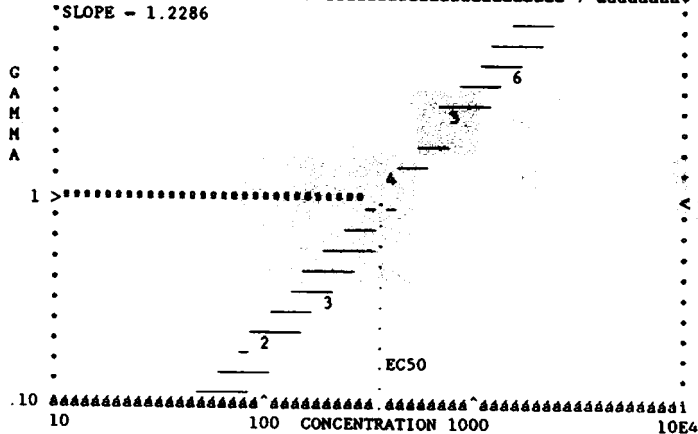
| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 77.00 | 48.186 | 0.2251 |
| 2 | 76.00 | 96.371 | 0.2412* |
| 3 | 68.00 | 192.743 | 0.3873* |
| 4 | 39.00 | 385.485 | 1.4188* |
| 5 | 25.00 | 770.970 | 2.7733* |
| 6 | 16.00 | 1541.941 | 4.8958* |
| 7 | 5.00 | 3083.881 | 17.8667* |
| 8 | 1.00 | 6167.763 | 93.3333 |
| 9 | 1.00 | 12335.530 | 93.3333 |
| 10 | 0.00 | 24671.051 | > 999 * |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

CONTROL It's : 95.00 92.00 96.00

```

10 ****SLOPE = 1.2286*****7 ****

```



ECSD 343.202 (95% CONFIDENCE RANGE: 272.486 TO 432.271)

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# Used for calculations
* Invalid gammas
```

FILE NAME: 5-1-4-3B.SPT

TEST TIME:

Room temp. 23.4°C

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

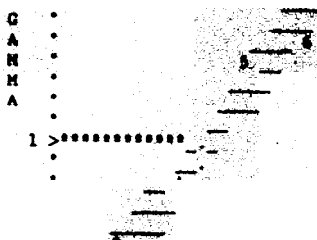
Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 95.00 | 48.186 | -0.0175* |
| 2 | 85.00 | 96.371 | 0.0980# |
| 3 | 67.00 | 192.743 | 0.3930# |
| 4 | 57.00 | 385.485 | 0.6374# |
| 5 | 26.00 | 770.970 | 2.5897# |
| 6 | 19.00 | 1541.941 | 3.9123# |
| 7 | 7.00 | 3083.881 | 12.3333# |
| 8 | 3.00 | 6167.763 | 30.1111 |
| 9 | 2.00 | 12335.530 | 45.6667 |
| 10 | 1.00 | 24671.051 | 92.3333 |
| 11 | 1.00 | 49342.102 | 92.3333 |
| 12 | 0.00 | 98684.203 | > 999 * |

| | | | |
|----------------|-------|-------|-------|
| CONTROL It's : | 97.00 | 97.00 | 86.00 |
|----------------|-------|-------|-------|

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*SLOPE = 1.3384



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EC50

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CONCENTRATION 10E4

EC50 475.297 (95% CONFIDENCE RANGE 380.706 TO 593.389)

Used for calculations
* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: PERCENT.SPT

TEST DATE:

Sample Description:

TEST TIME:

percent of 5-1-7-3A

Room temp: 22.4°C

Procedure: SOLID-PHASE

Osmotic Adjustment: no

Initial Concentration : 9.868 e

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: g

| NUMBER | It | CONC. | GAMMA |
|--------|--------|--------|-----------|
| 1 | 131.00 | 0.0048 | -0.0611* |
| 2 | 126.00 | 0.0096 | -0.0238* |
| 3 | 116.00 | 0.0193 | 0.0603 |
| 4 | 105.00 | 0.0385 | 0.1714 |
| 5 | 88.00 | 0.0771 | 0.3977# |
| 6 | 52.00 | 0.1542 | 1.3654# |
| 7 | 22.00 | 0.3084 | 4.5909# |
| 8 | 7.00 | 0.6168 | 16.5714# |
| 9 | 1.00 | 1.2335 | 122.0000* |
| 10 | 1.00 | 2.4670 | 122.0000* |
| 11 | 1.00 | 4.9340 | 122.0000* |
| 12 | 1.00 | 9.8680 | 122.0000* |

| | | | |
|----------------|-------|--------|--------|
| CONTROL It's : | 97.00 | 136.00 | 136.00 |
|----------------|-------|--------|--------|

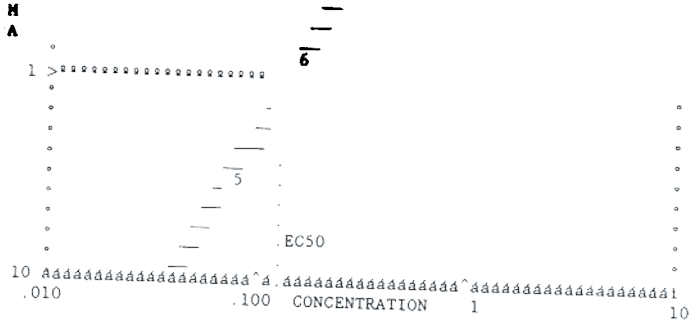
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10 ***** 8 *****
   *SLOPE = 1.7892

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*SLOPE = 1.7892

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0.1297 (95% CONFIDENCE RANGE: 0.1254 TO 0.1340)

Used for calculations
* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: 5-1-7-3A.SPT

TEST DATE: _____

TEST TIME: _____

Sample Description:

Room temp. 22.4°C

2:00pm, 1-13-93, Batchtown I and II, Detailed Test, Dupe A

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|-----------|
| 1 | 131.00 | 48.186 | -0.0611* |
| 2 | 126.00 | 96.371 | -0.0238* |
| 3 | 116.00 | 192.743 | 0.0603 |
| 4 | 105.00 | 385.485 | 0.1714 |
| 5 | 88.00 | 770.970 | 0.3977# |
| 6 | 52.00 | 1541.941 | 1.3654# |
| 7 | 22.00 | 3083.881 | 4.5909# |
| 8 | 7.00 | 6167.763 | 16.5714# |
| 9 | 1.00 | 12335.530 | 122.0000* |
| 10 | 1.00 | 24671.051 | 122.0000* |
| 11 | 1.00 | 49342.102 | 122.0000* |
| 12 | 1.00 | 98684.203 | 122.0000* |

| | | | | |
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| CONTROL | 1c's : | 97.00 | 136.00 | 136.00 |
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   *SLOPE = 1.7892

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*SLOPE = 1.7892

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Figure 1 is a log-log plot showing the relationship between the concentration of a chemical (X-axis) and the response (Y-axis). The X-axis is labeled 'CONCENTRATION' and ranges from 10 to 10⁵. The Y-axis ranges from 0 to 10. A horizontal line at Y=5 is labeled 'EC50'. The plot shows a sigmoidal curve with a horizontal line at Y=5 labeled 'EC50'.

EC50 1296.576 (95% CONFIDENCE RANGE: 1255.075 TO 1339.449)

Used for calculations
* Invalid gammas

MICROTOX DATA REPORT

FILE NAME: 5-1-7-3B.SPT

TEST DATE: _____

TEST TIME: _____

Sample Description:

Room temp 22.5

3:00pm, 1-13-93, Batchtown I and II, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 89.00 | 48.186 | 0.0524 |
| 2 | 80.00 | 96.371 | 0.1708 |
| 3 | 80.00 | 192.743 | 0.1708# |
| 4 | 67.00 | 385.485 | 0.3980# |
| 5 | 53.00 | 770.970 | 0.7673# |
| 6 | 32.00 | 1541.941 | 1.9271# |
| 7 | 12.00 | 3083.881 | 6.8056# |
| 8 | 5.00 | 6167.763 | 17.7333# |
| 9 | 2.00 | 12335.530 | 45.8333 |
| 10 | 1.00 | 24671.051 | 92.6667 |
| 11 | 1.00 | 49342.102 | 92.6667 |
| 12 | 1.00 | 98684.203 | 92.6667 |

```
CONTROL It's : 96.00 89.00 96.00
10 OAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAA 8 AAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAAA C
* SLOPE = 1.3458
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100 1000 CONCENTRATION 10E4 10E5

EC50 802.832 (95% CONFIDENCE RANGE 678.868 TO 949.432)

Used for calculation

MICROTOX DATA REPORT

FILE NAME: 5-1-11-A.SPT

TEST DATE: _____

TEST TIME: _____

Sample Description:

Room Temp: 21.9°C

2:00, 1-15-93, Batch town I and II, Detailed Test, Dupe A

Procedure: SOLID-PHASE

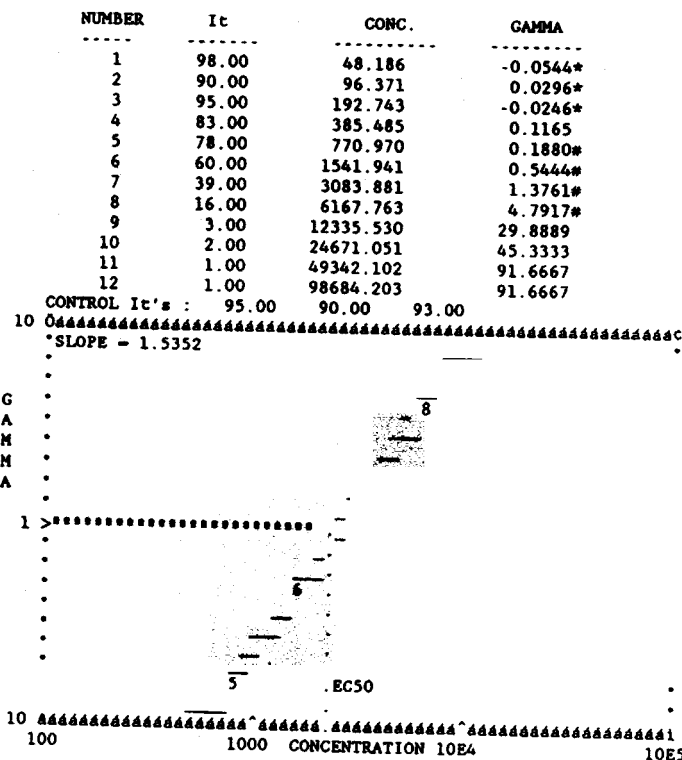
Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm



EC50 2324.261 (95% CONFIDENCE RANGE: 2027.790 TO 2664.076)

* Used for calculations
 * Invalid gammas

FILE NAME: 5-1-11-B.SPT

TEST DATE:

TEST TIME:

Sample Description:

Room temp. 21.9°C

2:40pm, 1-15-93, Batch town I and II, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

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EC50

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100                                1000 CONCENTRATION 10E4                                10E5

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EC50 3788.811 (95% CONFIDENCE RANGE: 3134.043 TO 4580.374)

```
# Used for calculations
* Invalid gammas
```

MICROTOX DATA REPORT

FILE NAME: 6-1-3-3A.SPT

TEST DATE: _____

TEST TIME:

Sample Description:

1:25pm, 2-12-93, Big Pond, Detailed Test, Dupe A

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

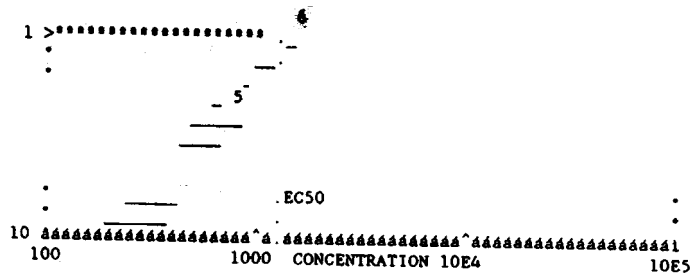
| NUMBER | It | CONC. | GAMMA |
|--------|-------|-----------|---------|
| 1 | 73.00 | 48.186 | 0.1644 |
| 2 | 73.00 | 96.371 | 0.1644 |
| 3 | 72.00 | 192.743 | 0.1806 |
| 4 | 65.00 | 385.485 | 0.3077 |
| 5 | 56.00 | 770.970 | 0.5179* |
| 6 | 37.00 | 1541.941 | 1.2973* |
| 7 | 21.00 | 3083.881 | 3.0476* |
| 8 | 5.00 | 6167.763 | 16.0000 |
| 9 | 3.00 | 12335.530 | 27.3333 |
| 10 | 3.00 | 24671.051 | 27.3333 |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

| | | | |
|----------------|-------|-------|-------|
| CONTROL It's : | 96.00 | 84.00 | 75.00 |
|----------------|-------|-------|-------|

10 *****
*SLOPE = 1.2785*****

*SLOPE = 1.2785

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EC50 1279.279 (95% CONFIDENCE RANGE: 1092.498 TO 1497.993)

```
# Used for calculations
* Invalid gammas
```

MICROTOX DATA REPORT

FILE NAME: 6-1-3-3B.SPT

TEST DATE: _____

Sample Description:

TEST TIME: _____

2:00pm, 2-12-93, Big Pond, Detailed Test, Dupe B

Room Temp. 21.9°C

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|----------|
| 1 | 0.00 | 48.186 | > 999 * |
| 2 | 139.00 | 96.371 | -0.7650* |
| 3 | 0.00 | 192.743 | > 999 * |
| 4 | 0.00 | 385.485 | > 999 * |
| 5 | 0.00 | 770.970 | > 999 * |
| 6 | 115.00 | 1541.941 | -0.7159* |
| 7 | 83.00 | 3083.881 | -0.6064* |
| 8 | 15.00 | 6167.763 | 1.1778 |
| 9 | 5.00 | 12335.530 | 5.5333 |
| 10 | 2.00 | 24671.051 | 15.3333 |
| 11 | 1.00 | 49342.102 | 31.6667 |
| 12 | 0.00 | 98684.203 | > 999 * |

CONTROL It's : 98.00 0.00 0.00

.....NO TOXICITY AT CONCENTRATIONS TESTED

* Invalid gammas

100

TEST DATE:

TEST TIME:

2:15pm, 2-10-93, Big Pond, Detailed Test, Dupe A

Osmotic Adjustment: none

Dilution Factor : 2

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|----------|
| 1 | 111.00 | 48.186 | -0.0991* |
| 2 | 100.00 | 96.371 | 0.0000* |
| 3 | 94.00 | 192.743 | 0.0638 |
| 4 | 90.00 | 385.485 | 0.1111 |
| 5 | 80.00 | 770.970 | 0.2500 |
| 6 | 64.00 | 1541.941 | 0.5625# |
| 7 | 32.00 | 3083.881 | 2.1250# |
| 8 | 5.00 | 6167.763 | 19.0000# |
| 9 | 1.00 | 12335.530 | 99.0000# |
| 10 | 0.00 | 24671.051 | > 999 * |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

| | | | |
|----------------|-------|--------|--------|
| CONTROL It's : | 95.00 | 101.00 | 104.00 |
|----------------|-------|--------|--------|

10 ***** 8 *** 9 *****
*SLOPE = 2.5539

**G
A
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M
A**

[illegible]

EC50 2060.982 (95% CONFIDENCE RANGE: 1539.959 TO 2758.287)

```
# Used for calculations
* Invalid gammas
```

FILE NAME: 6-1-4-3B.SPT

TEST TIME:

Sample Description:

3:10pm, 2-10-93, Big Pond, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|-----------|
| 1 | 110.00 | 48.186 | -0.0273* |
| 2 | 116.00 | 96.371 | -0.0776* |
| 3 | 112.00 | 192.743 | -0.0446* |
| 4 | 109.00 | 385.485 | -0.0183* |
| 5 | 98.00 | 770.970 | 0.0918# |
| 6 | 72.00 | 1541.941 | 0.4861# |
| 7 | 36.00 | 3083.881 | 1.9722# |
| 8 | 7.00 | 6167.763 | 14.2857# |
| 9 | 1.00 | 12335.530 | 106.0000* |
| 10 | 0.00 | 24671.051 | > 999 * |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

| | | | |
|----------------|-------|--------|--------|
| CONTROL It's : | 95.00 | 107.00 | 119.00 |
|----------------|-------|--------|--------|

[illegible]

*SLOPE - 2.3864

**G
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6

.LC50

```

10 #####^#####^#####1
100          1000 CONCENTRATION 10E4          10E5

```

EC50 2129.088 (95% CONFIDENCE RANGE: 1821.125 TO 2489.130)

Used for calculations
* Invalid games

FILE NAME: 6-2-1-3A.SPT

TEST TIME:

Room Temp.: 20.7°C

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|----------|
| 1 | 82.00 | 48.186 | 0.1545 |
| 2 | 100.00 | 96.371 | -0.0533* |
| 3 | 117.00 | 192.743 | -0.1909* |
| 4 | 97.00 | 385.485 | -0.0241* |
| 5 | 90.00 | 770.970 | 0.0519# |
| 6 | 73.00 | 1541.941 | 0.2968# |
| 7 | 49.00 | 3083.881 | 0.9320# |
| 8 | 29.00 | 6167.763 | 2.2644# |
| 9 | 4.00 | 12335.530 | 22.6667# |
| 10 | 1.00 | 24671.051 | 93.6667# |
| 11 | 0.00 | 49342.102 | > 999 * |
| 12 | 0.00 | 98684.203 | > 999 * |

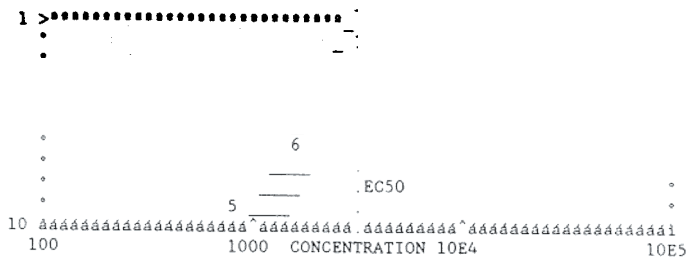
| | | | |
|----------------|-------|-------|-------|
| CONTROL It's : | 95.00 | 93.00 | 96.00 |
|----------------|-------|-------|-------|

```

10 ***** 9 *** 10 *****
   *SLOPE = 2.1183

```

**G
A
M
M**



EC50 3140.505 (95% CONFIDENCE RANGE: 2563.958 TO 3846.698)

```
# Used for calculations
* Invalid gammas
```

MICROTOX DATA REPORT

FILE NAME: 6-2-1-3B.SPT

TEST DATE: _____

Sample Description:

TEST TIME: _____
Ran Temp.: 21.9°C

1:15pm, 2-12-93, Bryrants Creek, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|-----------------------------------|--------|-----------|----------|
| 1 | 113.00 | 48.186 | -0.2153* |
| 2 | 108.00 | 96.371 | -0.1790* |
| 3 | 104.00 | 192.743 | -0.1474* |
| 4 | 111.00 | 385.485 | -0.2012* |
| 5 | 0.00 | 770.970 | > 999 * |
| 6 | 80.00 | 1541.941 | 0.1083# |
| 7 | 49.00 | 3083.881 | 0.8095# |
| 8 | 30.00 | 6167.763 | 1.9556# |
| 9 | 7.00 | 12335.530 | 11.6667# |
| 10 | 2.00 | 24671.051 | 43.3333# |
| 11 | 1.00 | 49342.102 | 87.6667# |
| 12 | 1.00 | 98684.203 | 87.6667 |
| CONTROL It's : 96.00 53.00 117.00 | | | |

10 0444444444444444 5 4444444444444444 9 444 10 44 11 4444
 *SLOPE = 0.3538

G
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1 >*****

10 *****
 100 1000 CONCENTRATION 10E4 10E5
 .EC50
 6

EC50 842.851 (95% CONFIDENCE RANGE 19.604 TO 36237.098)

Used for calculations
 * Invalid gammas

FILE NAME: 6-2-2-3A.SPT

TEST DATE:

TEST TIME:

Room Temp.: 21.9°C

Sample Description:

2:15pm, 2-12-93, Bryrant's Creek, Detailed Test, Dupe A

Procedure: SOLID-PHASE

Initial Concentration : 98684.2 ppm

Test Time: 5 minutes

Osmotic Adjustment: none

Dilution Factor : 2

Concentration Units: ppm

| NUMBER | It | CONC. | GAMMA |
|--------|--------|-----------|-----------|
| 1 | 116.00 | 48.186 | -0.0776* |
| 2 | 110.00 | 96.371 | -0.0273* |
| 3 | 99.00 | 192.743 | 0.0808 |
| 4 | 75.00 | 385.485 | 0.4267 |
| 5 | 0.00 | 770.970 | > 999 * |
| 6 | 86.00 | 1541.941 | 0.2442* |
| 7 | 56.00 | 3083.881 | 0.9107* |
| 8 | 27.00 | 6167.763 | 2.9630* |
| 9 | 13.00 | 12335.530 | 7.2308* |
| 10 | 3.00 | 24671.051 | 34.6667 |
| 11 | 1.00 | 49342.102 | 106.0000* |
| 12 | 1.00 | 98684.203 | 106.0000* |

CONTROL It's : 96.00 105.00 120.00

*SLOPE = 1.6366

10

9

8

7

6

5

4

3

2

1

EC50

100 1000 CONCENTRATION 10E4 10E5

EC50 3441.762 (95% CONFIDENCE RANGE: 2795.067 TO 4238.083)

```
# Used for calculations
* Invalid gammas
```

FILE NAME: 6-2-2-3B.SPT

TEST DATE: _____
TEST TIME: _____

Room temp. 21.9°C

2:40pm, 2-12-93, Bryant's Creek, Detailed Test, Dupe B

Procedure: SOLID-PHASE

Osmotic Adjustment: none

Initial Concentration : 98684.2 ppm

Osmotic Adjustment:
Dilution Factor : 2

Test Time: 5 minutes

Concentration Units: ppm

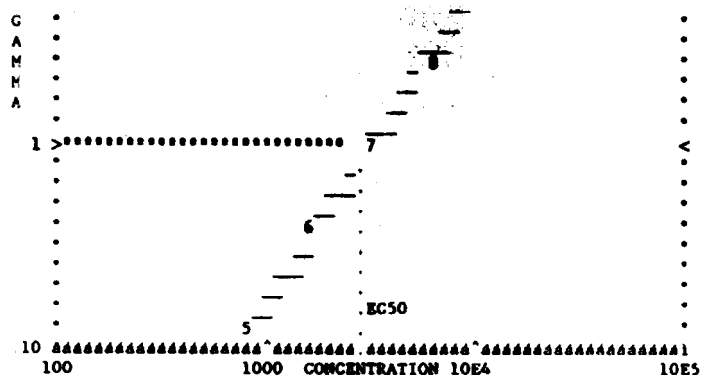
| NUMBER | 1t | CONC. | GAMMA |
|--------|-------|-----------|----------|
| 1 | 55.00 | 48.186 | -0.0061* |
| 2 | 53.00 | 96.371 | 0.0314* |
| 3 | 57.00 | 192.743 | -0.0409* |
| 4 | 48.00 | 385.485 | 0.1389 |
| 5 | 49.00 | 770.970 | 0.1156# |
| 6 | 39.00 | 1541.941 | 0.4017# |
| 7 | 26.00 | 3083.881 | 1.1026# |
| 8 | 15.00 | 6167.763 | 2.6444# |
| 9 | 5.00 | 12335.530 | 9.9333# |
| 10 | 2.00 | 24671.051 | 26.3333# |
| 11 | 1.00 | 49342.102 | 53.6667 |
| 12 | 0.00 | 98684.203 | > 999 * |

| | | | |
|-----------------------|--------------|--------------|--------------|
| CONTROL It's : | 95.00 | 16.00 | 53.00 |
|-----------------------|--------------|--------------|--------------|

```

10 *****SLOPE = 1.5515*****9*****

```



EC50 2975.663 (95% CONFIDENCE RANGE: 2729.961 TO 3243.478)

Used for calculations
* Invalid games

MICROTOX DATA REPORT

FILE NAME: 3-WINTER.SPT

3-1-148C

Sample Description:

TEST DATE:

TEST TIME:

Room temp. 22.2°C

3:20pm, 2-10-93, Keithsburg (Winter), Detailed Test, Dupe A

Procedure: SOLID-PHASE

Initial Concentration : 98684.2 ppm

Test Time: 5 minutes

Osmotic Adjustment: none

Dilution Factor : 2

Concentration Units: ppm

| | NUMBER | It | CONC. | GAMMA |
|-----------------|------------------------------------|-------|-----------|----------|
| | 1 | 92.00 | 48.186 | 0.0036* |
| | 2 | 39.00 | 96.371 | 1.3675# |
| | 3 | 76.00 | 192.743 | 0.2149# |
| | 4 | 65.00 | 385.485 | 0.4205# |
| | 5 | 34.00 | 770.970 | 1.7157# |
| | 6 | 21.00 | 1541.941 | 3.3968# |
| | 7 | 9.00 | 3083.881 | 9.2593# |
| | 8 | 4.00 | 6167.763 | 22.0833# |
| | 9 | 4.00 | 12335.530 | 22.0833# |
| | 10 | 2.00 | 24671.051 | 45.1667# |
| | 11 | 2.00 | 49342.102 | 45.1667# |
| | 12 | 1.00 | 98684.203 | 91.3333# |
| CONTROL It's : | 93.00 | 96.00 | 88.00 | |
| 10 | ***** 8 *** 9 *** 10 ** 11 ** 12 * | | | |
| *SLOPE = 0.8317 | | | | |

$$1 > \frac{2}{\sqrt{10}}$$

```

      .          .EC50          .
      .          .          .
10  ^^^^^^^^^^^^^^^^^^ ^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^
100          1000  CONCENTRATION 10E4          10E5

```

EC50 436.952 (95% CONFIDENCE RANGE: 201.172 TO 949.073)

```
# Used for calculations
* Invalid gammas
```

Appendix E

Sediment Elutriate Toxicity Test Results



UNIVERSITY HYGIENIC LABORATORY
TOXICITY TEST

DML Lab #2261003

Keithsburg Division

Site Mark Twain MNR

Source Lab Replicate #1

Sample Type sediment

Date Collected 7/14/92

Date Received 7/22/92

Test Organism Pimephales promelas

Age 9 days

Reference Toxicant NaCl

IC₅₀ 5.73

Chemical and Toxicity Test Data

Test Begun on 8/3/92 at 930 hrs

Test Ended on 8/7/92 at 930 hrs

| | Initial | 48 hours | 96 hours |
|----------------------------------|---------|----------|----------|
| NH ₃ -N (mg/L) | 3.8 | 3.1 | 2.0 |
| Total NH ₃ (mg/L) | 4.6 | 3.8 | 2.4 |
| Unionized NH ₃ (mg/L) | | 0.43 | |

| CHEMICAL AND PHYSICAL ANALYSIS | | | | FATHEAD MINNOW MORTALITY* | | | | | |
|--------------------------------|-----------|-----|-----|---------------------------|-----|-----|---------|-----|-----|
| | | | | SAMPLE | | | CONTROL | | |
| Time | Temp (°C) | DO | pH | a | b | c | a | b | c |
| Initial | 24.0 | 5.1 | 7.7 | | | | | | |
| 24 hours | 26.0 | 8.1 | 8.3 | | | | | | |
| 48 hours | 27.0 | 8.0 | 8.3 | | | | | | |
| 72 hours | 27.0 | 8.6 | 8.1 | | | | | | |
| 96 hours | 25.0 | 7.5 | 8.1 | 0/7 | 0/7 | 0/6 | 0/7 | 0/7 | 0/6 |
| TOTAL MORTALITY AT 96 HOURS | | | | 0/20 | | | 0/20 | | |

* Mortality = Number Dead / Number Tested

Comments:

Analyst: JLS/JGM
Date Reported: AUG 11 1992

Verified: *pk*



UNIVERSITY HYGIENIC LABORATORY
TOXICITY TEST

DML Lab # 9261003

Keithsburg Division
Site Mark Twain NWR Source Lab Replicate #2

Sample Type sediment Date Collected 7/14/92 Date Received 7/22/92

Test Organism Pimephales promelas Age 9 days Reference Toxicant NaCl

LC₅₀ 5.73

Chemical and Toxicity Test Data

Test Begun on 8/3/92 at 930 hrs

Test Ended on 8/7/92 at 930 hrs

| | Initial | 48 hours | 96 hours |
|----------------------------------|---------|----------|----------|
| NH ₃ -N (mg/L) | 3.7 | 3.2 | 1.6 |
| Total NH ₃ (mg/L) | 4.5 | 3.9 | 1.9 |
| Unionized NH ₃ (mg/L) | 0.12 | | 0.20 |

| CHEMICAL AND PHYSICAL ANALYSIS | | | | FATHEAD MINNOW MORTALITY* | | | | | |
|--------------------------------|-----------|-----|-----|---------------------------|-----|-----|---------|-----|-----|
| | | | | SAMPLE | | | CONTROL | | |
| Time | Temp (°C) | DO | pH | a | b | c | a | b | c |
| Initial | 24.0 | 5.0 | 7.7 | | | | | | |
| 4 hours | 26.0 | 8.0 | 8.4 | | | | | | |
| 48 hours | 25.0 | 8.1 | 8.3 | | | | | | |
| 72 hours | 27.0 | 8.5 | 8.3 | | | | | | |
| 96 hours | 25.0 | 7.7 | 8.3 | 0/7 | 0/7 | 0/6 | 0/7 | 1/7 | 0/6 |
| TOTAL MORTALITY AT 96 HOURS | | | | 0/20 | | | 1/20 | | |

* Mortality = Number Dead / Number Tested

Comments:

Analyst: JLS/JGM
Date Reported: AUG 11 1992

Verified: *gjh*



UNIVERSITY HYGIENIC LABORATORY
TOXICITY TEST

DML Lab # 9264461

Site Keithsburg Division MT-3-5-2-2 Source Lab Replicate #1

Sample Type Sediment Date Collected 9/14/92 Date Received 9/17/92

Test Organism Pimephales promelas Age 16 days Reference Toxicant NaCl

LC₅₀ 5.73 (48 hour)

Chemical and Toxicity Test Data

Test Begun on 9/21/92 at 1030 hrs

Test Ended on 9/23/92 at 1030 hrs

| | Initial | 48 hours | 96 hours |
|----------------------------------|-------------|------------|-------------|
| NH ₃ -N (mg/L) | <u>8.7</u> | <u>7.2</u> | <u>2.6</u> |
| Total NH ₃ (mg/L) | <u>10.6</u> | <u>8.7</u> | <u>3.2</u> |
| Unionized NH ₃ (mg/L) | <u>0.27</u> | <u>1.0</u> | <u>0.31</u> |

| CHEMICAL AND PHYSICAL ANALYSIS | | | | FATHEAD MINNOW MORTALITY* | | | | | |
|--------------------------------|-----------|-----|-----|---------------------------|-----|-----|---------|-----|-----|
| | | | | SAMPLE | | | CONTROL | | |
| Time | Temp (°C) | DO | pH | a | b | c | a | b | c |
| Initial | 23.0 | 5.5 | 7.7 | | | | | | |
| 24 hours | 24.0 | 7.9 | 8.4 | | | | | | |
| 48 hours | 24.0 | 8.6 | 8.4 | | | | | | |
| hours | 24.0 | 7.2 | 8.3 | | | | | | |
| 96 hours | 25.0 | 7.3 | 8.3 | 3/7 | 1/7 | 0/6 | 0/7 | 0/7 | 0/6 |
| TOTAL MORTALITY AT 96 HOURS | | | | 4/20 | | | 0/20 | | |

* Mortality = Number Dead / Number Tested

Comments:

Analyst: JLS/JGM
Date Reported:

Verified: gfk



UNIVERSITY HYGIENIC LABORATORY
TOXICITY TEST

DML Lab # 9264461

Site Keithsburg Division MT-3-5-2-2 Source Lab Replicate #2

Sample Type Sediment Date Collected 9/14/92 Date Received 9/17/92

Test Organism Pimephales promelas Age 16 days Reference Toxicant NaCl

IC₅₀ 5.73 (48 hour)

Chemical and Toxicity Test Data

Test Begun on 9/21/92 at 1030 hrs

Test Ended on 9/25/92 at 1030 hrs

| | Initial | 48 hours | 96 hours |
|----------------------------------|-------------|------------|-------------|
| NH ₃ -N (mg/L) | <u>9.2</u> | <u>7.2</u> | <u>2.8</u> |
| Total NH ₃ (mg/L) | <u>11.2</u> | <u>8.7</u> | <u>3.4</u> |
| Unionized NH ₃ (mg/L) | <u>0.22</u> | <u>0</u> | <u>0.35</u> |

| CHEMICAL AND PHYSICAL ANALYSIS | | | | FATHEAD MINNOW MORTALITY* | | | | | |
|--------------------------------|-----------|-----|-----|---------------------------|-----|-----|---------|-----|-----|
| | | | | SAMPLE | | | CONTROL | | |
| Time | Temp (°C) | DO | pH | a | b | c | a | b | c |
| Initial | 23.0 | 5.5 | 7.6 | | | | | | |
| 24 hours | 24.0 | 7.9 | 8.4 | | | | | | |
| 48 hours | 24.0 | 8.3 | 8.4 | | | | | | |
| 72 hours | 24.0 | 7.2 | 8.4 | | | | | | |
| 96 hours | 25.0 | 7.1 | 8.3 | 1/7 | 2/7 | 1/6 | 0/7 | 0/7 | 0/6 |
| TOTAL MORTALITY AT 96 HOURS | | | | 4/20 | | | 0/20 | | |

* Mortality = Number Dead / Number Tested

Comments:

Analyst: JLS/JGM
Date Reported:

Verified JK



UNIVERSITY HYGIENIC LABORATORY
TOXICITY TEST

DML Lab # 9264462

Site Keithsburg Division MT-3-5-3-2 Source Lab Replicate #1

Sample Type Sediment Date Collected 9/14/92 Date Received 9/17/92

Test Organism Pimephales promelas Age 16 days Reference Toxicant NaCl

LC₅₀ 5.73 (48 hour)

Chemical and Toxicity Test Data

Test Begun on 9/21/92 at 1030 hrs

Test Ended on 9/23/92 at 1030 hrs

| | Initial | 48 hours | 96 hours |
|----------------------------------|-------------|-------------|-------------|
| NH ₃ -N (mg/L) | <u>7.8</u> | <u>6.4</u> | <u>2.7</u> |
| Total NH ₃ (mg/L) | <u>9.5</u> | <u>7.8</u> | <u>3.1</u> |
| Unionized NH ₃ (mg/L) | <u>0.18</u> | <u>0.92</u> | <u>0.34</u> |

| CHEMICAL AND PHYSICAL ANALYSIS | | | | FATHEAD MINNOW MORTALITY* | | | | | |
|--------------------------------|-----------|-----|-----|---------------------------|-----|-----|---------|-----|-----|
| | | | | SAMPLE | | | CONTROL | | |
| Time | Temp (°C) | DO | pH | a | b | c | a | b | c |
| Initial | 23.0 | 5.6 | 7.6 | | | | | | |
| 24 hours | 24.0 | 8.0 | 8.4 | | | | | | |
| 48 hours | 24.0 | 8.4 | 8.4 | | | | | | |
| 72 hours | 24.0 | 7.1 | 8.2 | | | | | | |
| 96 hours | 25.0 | 7.2 | 8.3 | 0/7 | 2/7 | 2/6 | 0/7 | 0/7 | 0/6 |
| TOTAL MORTALITY AT 96 HOURS | | | | 4/20 | | | 0/20 | | |

* Mortality = Number Dead / Number Tested

Comments:

Analyst: JLS/JGM
Date Reported:

Verified: *JK*



UNIVERSITY HYGIENIC LABORATORY
TOXICITY TEST

DHL Lab # 9264462

Site Keithsburg Division MT-3-5-3-2 Source Lab Replicate #2

Sample Type Sediment Date Collected 9/14/92 Date Received 9/17/92

Test Organism Pimephales promelas Age 16 days Reference Toxicant NaCl

LC₅₀ 5.73 (48 hour)

Chemical and Toxicity Test Data

Test Begun on 9/21/92 at 1030 hrs

Test Ended on 9/25/92 at 1030 hrs

| | Initial | 48 hours | 96 hours |
|----------------------------------|-------------|------------|-------------|
| NH ₃ -N (mg/L) | <u>8.3</u> | <u>6.6</u> | <u>2.2</u> |
| Total NH ₃ (mg/L) | <u>10.1</u> | <u>8.0</u> | <u>2.7</u> |
| Unionized NH ₃ (mg/L) | <u>0.19</u> | <u>1.2</u> | <u>0.34</u> |

| CHEMICAL AND PHYSICAL ANALYSIS | | | | FATHEAD MINNOW MORTALITY* | | | | | |
|--------------------------------|-----------|-----|-----|---------------------------|-----|-----|---------|-----|-----|
| | | | | SAMPLE | | | CONTROL | | |
| Time | Temp (°C) | DO | pH | a | b | c | a | b | c |
| Initial | 23.0 | 5.6 | 7.6 | | | | | | |
| 24 hours | 24.0 | 8.3 | 8.4 | | | | | | |
| 48 hours | 24.0 | 8.4 | 8.5 | | | | | | |
| 72 hours | 24.0 | 7.6 | 8.4 | | | | | | |
| 96 hours | 25.0 | 7.7 | 8.4 | 1/7 | 0/7 | 0/6 | 0/7 | 0/7 | 0/6 |
| TOTAL MORTALITY AT 96 HOURS | | | | 1/20 | | | 0/20 | | |

* Mortality = Number Dead / Number Tested

Comments:

Analyst: JLS/JGM
Date Reported:

Verified: JK



UNIVERSITY HYGIENIC LABORATORY
TOXICITY TEST

DHL Lab #

Site Control Source Lab Replicate # 1

Sample Type water Date Collected Date Received

Test Organism Pimephales promelas Age 9 days Reference Toxicant NaCl

LC₅₀ 5.73

Chemical and Toxicity Test Data

Test Begun on 8/3/92 at 930 hrs

Test Ended on 8/7/92 at 930 hrs

| | Initial | 48 hours | 96 hours |
|----------------------------------|-----------------|-------------|-------------|
| NH ₃ -N (mg/L) | <u><0.1</u> | <u>0.2</u> | <u>0.2</u> |
| Total NH ₃ (mg/L) | <u><0.1</u> | <u>0.2</u> | <u>0.2</u> |
| Unionized NH ₃ (mg/L) | <u><0.01</u> | <u>0.02</u> | <u>0.02</u> |

| CHEMICAL AND PHYSICAL ANALYSIS | | | | FATHEAD MINNOW MORTALITY* | | | | | |
|--------------------------------|-----------|------|-----|---------------------------|---|---|---------|-----|-----|
| | | | | SAMPLE | | | CONTROL | | |
| Time | Temp (°C) | DO | pH | a | b | c | a | b | c |
| Initial | 24.0 | 11.2 | 8.3 | | | | | | |
| 24 hours | 26.0 | 8.3 | 8.3 | | | | | | |
| 48 hours | 26.0 | 8.4 | 8.3 | | | | | | |
| 72 hours | 27.0 | 8.8 | 8.2 | | | | | | |
| 96 hours | 25.0 | 8.2 | 8.3 | | | | 0/7 | 0/7 | 0/6 |
| TOTAL MORTALITY AT 96 HOURS | | | | | | | 0/20 | | |

* Mortality = Number Dead / Number Tested

Comments:

Analyst: JLS/JGM
Date Reported: AUG 11 1992

Verified: *gh*



UNIVERSITY HYGIENIC LABORATORY
TOXICITY TEST

DML Lab #

Control Source Lab Replicate # 2

Sample Type water Date Collected Date Received

Test Organism Pimephales promelas Age 9 days Reference Toxicant NaCl

LC₅₀ 5.73

Chemical and Toxicity Test Data

Test Begun on 8/3/92 at 930 hrs

Test Ended on 8/7/92 at 930 hrs

| | Initial | 48 hours | 96 hours |
|----------------------------------|---------|----------|----------|
| NH ₃ -N (mg/L) | <0.1 | 0.1 | 0.3 |
| Total NH ₃ (mg/L) | <0 | 0.1 | 0.4 |
| Unionized NH ₃ (mg/L) | <0.01 | 0.01 | 0.04 |

| CHEMICAL AND PHYSICAL ANALYSIS | | | | FATHEAD MINNOW MORTALITY* | | | | | |
|--------------------------------|-----------|------|-----|---------------------------|---|---|---------|-----|-----|
| | | | | SAMPLE | | | CONTROL | | |
| Time | Temp (°C) | DO | pH | a | b | c | a | b | c |
| Initial | 24.0 | 11.0 | 8.3 | | | | | | |
| 24 hours | 26.0 | 8.4 | 8.3 | | | | | | |
| 48 hours | 27.0 | 8.5 | 8.2 | | | | | | |
| 72 hours | 25.0 | 8.9 | 8.2 | | | | | | |
| 96 hours | 25.0 | 8.2 | 8.3 | | | | 0/7 | 1/7 | 0/6 |
| TOTAL MORTALITY AT 96 HOURS | | | | | | | 1/26 | | |

* Mortality = Number Dead / Number Tested

Comments:

Analyst: JLS/JCM
Date Reported: AUG 11 1992

Verified

Appendix F

Water Quality Data Records

Appendix F. Water quality data for the sampling locations at Mark Twain National Wildlife Refuge study area, 1992.

| SITE | REFERENCE NUMBER | DATE | TIME | WATER DEPTH (meters) | WATER TEMP (celsius) | CONDUCTIVITY (uS/cm) | DO-top (mg/L) | DO-bottom (mg/L) |
|-----------------------|---------------------|-----------|------|----------------------------|----------------------------|-------------------------|------------------|---------------------|
| Stunk Slough | 1 | 9-Jun-92 | 1200 | 1.00 | 27.00 | 440.00 | 6.20 | |
| | 2 | 9-Jun-92 | 1300 | 2.50 | 27.00 | 470.00 | 8.00 | |
| | 3 | 9-Jun-92 | 1500 | 2.50 | 26.00 | 440.00 | 10.00 | |
| Big Timber Division | 4 | 22-May-92 | 1030 | 8.50 | 23.00 | 310.00 | 7.50 | |
| | 5 | 22-May-92 | 1130 | 2.00 | 25.00 | 320.00 | 11.00 | |
| | 6 | 22-May-92 | 1300 | 4.00 | | | | |
| | 7 | 22-May-92 | 1400 | 3.00 | | | | |
| | 8 | 22-May-92 | 1500 | 3.00 | | | | |
| | 9 | 11-Jun-92 | 930 | 0.50 | 23.50 | 400.00 | 8.30 | |
| | 10 | 11-Jun-92 | 1100 | 0.90 | 25.00 | 400.00 | 11.00 | |
| | 11 | 11-Jun-92 | 1300 | 3.50 | 22.00 | 390.00 | 11.20 | 5.80 |
| Keithsburg Division | 12 | 11-Jun-92 | 1500 | 4.00 | 23.00 | 370.00 | 14.00 | 6.20 |
| | 13 | 19-May-92 | 1030 | 5.00 | 21.20 | 340.00 | 5.20 | 3.50 |
| | 14 | 19-May-92 | 1130 | 5.50 | 21.20 | 340.00 | 5.30 | 3.40 |
| | 15 | 19-May-92 | 1300 | 2.75 | 20.50 | 340.00 | 15.00 | 10.20 |
| | 16 | 19-May-92 | 1400 | 2.75 | 20.20 | 310.00 | 15.00 | 11.30 |
| | 17 | 19-May-92 | 1530 | 5.25 | 22.50 | | 13.00 | 12.00 |
| | 18 | 14-Jul-92 | 1000 | 3.50 | 25.50 | 280.00 | 4.10 | 0.50 |
| | 19 | 14-Jul-92 | 1200 | 4.00 | 26.00 | 315.00 | 4.40 | 3.60 |
| | 13/14/17a | 14-Sep-92 | 1000 | 2.50 | 19.00 | 430.00 | 2.00 | 1.00 |
| | 13/14/17b | 14-Sep-92 | 1030 | 2.50 | 19.00 | 455.00 | 2.20 | 0.80 |
| | 13/14/17c | 14-Sep-92 | 1100 | 2.50 | 20.00 | 470.00 | 2.00 | 1.20 |
| | 13/14/17d | 14-Sep-92 | 1130 | 2.50 | 20.00 | 350.00 | 4.20 | 3.80 |
| | 13/14/17e | 14-Sep-92 | 1200 | 2.50 | 20.00 | 350.00 | 5.70 | 4.00 |
| Gardner Division | 20 | 18-May-92 | 1000 | 1.75 | 19.50 | 310.00 | 8.10 | |
| | 21 | 18-May-92 | 1100 | 1.75 | | | | |
| | 22 | 18-May-92 | 1130 | 1.50 | 20.50 | 355.00 | 8.30 | |
| | 23 | 18-May-92 | 1200 | 1.75 | 21.50 | 455.00 | 9.20 | |
| Clarence Cannon Refug | 24 | 14-May-92 | | 1.75 | | | | |
| | 25 | 14-May-92 | | 2.00 | 19.00 | 190.00 | 10.50 | |
| | 26 | 14-May-92 | | 3.00 | | | | |
| | 27 | 14-May-92 | | 3.00 | 19.00 | 200.00 | 11.00 | |
| | 28 | 14-May-92 | | 4.00 | 18.00 | 430.00 | 7.50 | |
| | 29 | 14-May-92 | | 3.50 | | | | |
| | 30 | 14-May-92 | | 5.50 | 17.00 | 290.00 | 12.00 | |
| | 31 | 14-May-92 | | 11.00 | 17.00 | 280.00 | 12.00 | |
| Batchtown Division | 32 | 12-May-92 | 1000 | 4.00 | 21.00 | 285.00 | 13.80 | |
| | 33 | 12-May-92 | 1015 | 4.00 | | | | |
| | 34 | 12-May-92 | 1035 | 4.50 | 21.50 | 295.00 | 11.70 | 9.90 |
| | 35 | 12-May-92 | 1045 | 4.50 | 21.50 | 295.00 | 15.00 | 13.40 |
| | 36 | 12-May-92 | 1130 | 4.50 | | | | |
| | 37 | 12-May-92 | 1200 | 4.00 | 21.00 | 319.00 | 14.60 | |
| | 38 | 12-May-92 | 1300 | 4.50 | 23.00 | 310.00 | 15.00 | 15.00 |
| | 39 | 12-May-92 | 1330 | 2.50 | | | | |
| | 40 | 13-May-92 | 1200 | 2.50 | 20.50 | 250.00 | 9.70 | |
| | 41 | 13-May-92 | 1230 | 3.00 | 21.00 | 310.00 | 13.50 | |
| | 42 | 13-May-92 | 1245 | 3.50 | 21.00 | 350.00 | 13.00 | |
| | 43 | 13-May-92 | 1410 | 4.00 | 17.50 | 335.00 | 5.40 | 1.00 |

Appendix G

Quality Assurance and Quality Control Test Results

PROCEDURAL BLANKS

| Analyte | Lab Sample Number | Result Total UG |
|------------|-------------------|-----------------|
| % Moisture | BLANK-B | |
| | BLANK-F | |
| | BLANK-G | |
| | BLANK-J | |
| | BLANK-N | |
| | BLANK-P | |
| Al | BLANK-B | -47 |
| | BLANK-F | 4 |
| | BLANK-G | 4 |
| | BLANK-J | 26 |
| | BLANK-N | 8 |
| | BLANK-P | 4 |
| As | BLANK-B | -.02 |
| | BLANK-F | .01 |
| | BLANK-G | -.02 |
| | BLANK-J | -.01 |
| | BLANK-N | .03 |
| | BLANK-P | -.01 |
| Cd | BLANK-B | 0 |
| | BLANK-F | 0 |
| | BLANK-G | 0 |
| | BLANK-J | 0 |
| | BLANK-N | 0 |
| | BLANK-P | 0 |
| Cr | BLANK-B | -.5 |
| | BLANK-F | .04 |
| | BLANK-G | -1.09 |
| | BLANK-J | 1.5 |
| | BLANK-N | .01 |
| | BLANK-P | .03 |

PROCEDURAL BLANKS (Cont.)

| Analyte | Lab Sample Number | Result Total UG |
|---------|-------------------|-----------------|
| Pb | BLANK-B | .07 |
| | BLANK-F | .02 |
| | BLANK-G | .02 |
| | BLANK-J | 0 |
| | BLANK-N | .03 |
| | BLANK-P | .02 |
| Se | BLANK-B | .01 |
| | BLANK-F | 0 |
| | BLANK-G | -.02 |
| | BLANK-J | .03 |
| | BLANK-N | 0 |
| | BLANK-P | 0 |
| Zn | BLANK-B | -.9 |
| | BLANK-F | -8.1 |
| | BLANK-G | -7.9 |
| | BLANK-J | 7.4 |
| | BLANK-N | 4.7 |
| | BLANK-P | 4.8 |

DUPLICATES

| Analyte | Sample Number | Sample Matrix | Initial Result (ppm / %) | Duplicate Result (ppm / %) | Relative % Average | Difference |
|------------|---------------|---------------|-----------------------------|-------------------------------|-----------------------|------------|
| % Moisture | 0-1-1-1 | Sediments | 38.9 % | 43.3 % | 41.1 | 10.71 |
| | 3-2-1-1 | Sediments | 52.8 % | 53.4 % | 53.1 | 1.13 |
| | 4-1-3-2 | Sediments | 36.9 % | 37.3 % | 37.1 | 1.08 |
| | 5-1-3-1 | Sediments | 51.2 % | 46.1 % | 48.65 | 10.48 |
| | 6-1-4-1 | Sediments | 49.9 % | 49.3 % | 49.6 | 1.21 |
| | 7-1-3-1 | Sediments | 31 % | 30.7 % | 30.85 | 0.97 |
| AVS | 0-1-1-1 | Sediments | 564.7 Dry | 603 Dry | 583.85 | 6.56 |
| | 3-2-1-1 | Sediments | 377 Dry | 391.8 Dry | 384.4 | 3.85 |
| | 4-1-3-2 | Sediments | 174.7 Dry | 155.4 Dry | 165.05 | 11.69 |
| | 5-1-3-1 | Sediments | 805.9 Dry | 743.3 Dry | 774.6 | 8.08 |
| | 6-1-4-1 | Sediments | 103.4 Dry | 99.3 Dry | 101.35 | 4.05 |
| | 7-1-3-1 | Sediments | 27.5 Dry | 28.1 Dry | 27.8 | 2.16 |
| Al | 0-1-1-1 | Sediments | 19917 Dry | 23788 Dry | 21852.5 | 17.71 |
| | 3-2-1-1 | Sediments | 12626 Dry | 11696 Dry | 12161 | 7.65 |
| | 4-1-3-2 | Sediments | 7967 Dry | 11578 Dry | 9772.5 | 36.95 |
| | 5-1-3-1 | Sediments | 22142 Dry | 21039 Dry | 21590.5 | 5.11 |
| | 6-1-4-1 | Sediments | 8046 Dry | 10306 Dry | 9176 | 24.63 |
| | 7-1-3-1 | Sediments | 7261 Dry | 10695 Dry | 8978 | 38.25 |
| As | 0-1-1-1 | Sediments | 5.84 Dry | 7.43 Dry | 6.635 | 23.96 |
| | 3-2-1-1 | Sediments | 6.54 Dry | 7.05 Dry | 6.795 | 7.51 |
| | 4-1-3-2 | Sediments | 4.24 Dry | 4.46 Dry | 4.35 | 5.06 |
| | 5-1-3-1 | Sediments | 5.64 Dry | 5.48 Dry | 5.56 | 2.88 |
| | 6-1-4-1 | Sediments | 3.06 Dry | 3.05 Dry | 3.055 | 0.33 |
| | 7-1-3-1 | Sediments | 5.3 Dry | 5.63 Dry | 5.465 | 6.04 |
| Cd | 0-1-1-1 | Sediments | .38 Dry | .42 Dry | 0.4 | 10 |
| | 3-2-1-1 | Sediments | .58 Dry | .63 Dry | 0.605 | 8.26 |
| | 4-1-3-2 | Sediments | .41 Dry | .41 Dry | 0.41 | 0 |
| | 5-1-3-1 | Sediments | .52 Dry | .56 Dry | 0.54 | 7.41 |
| | 6-1-4-1 | Sediments | .35 Dry | .38 Dry | 0.365 | 8.22 |
| | 7-1-3-1 | Sediments | .33 Dry | .35 Dry | 0.34 | 5.88 |

DUPLICATES (Cont.)

| Analyte | Sample Number | Sample Matrix | Initial Result (ppm / %) | Duplicate Result (ppm / %) | Relative % Average | Difference |
|---------|---------------|---------------|-----------------------------|-------------------------------|-----------------------|------------|
| Cr | 0-1-1-1 | Sediments | 18.91 Dry | 23.3 Dry | 21.105 | 20.8 |
| | 3-2-1-1 | Sediments | 24.27 Dry | 17.99 Dry | 21.13 | 29.72 |
| | 4-1-3-2 | Sediments | 10.64 Dry | 14.21 Dry | 12.425 | 28.73 |
| | 5-1-3-1 | Sediments | 28.16 Dry | 27.02 Dry | 27.59 | 4.13 |
| | 6-1-4-1 | Sediments | 7.91 Dry | 10.08 Dry | 8.995 | 24.12 |
| | 7-1-3-1 | Sediments | 9.65 Dry | 12.75 Dry | 11.2 | 27.68 |
| Cu | 0-1-1-1 | Sediments | 26.06 Dry | 31.27 Dry | 28.665 | 18.18 |
| | 3-2-1-1 | Sediments | 20.54 Dry | 22.94 Dry | 21.74 | 11.04 |
| | 4-1-3-2 | Sediments | 12.77 Dry | 12.89 Dry | 12.83 | 0.94 |
| | 5-1-3-1 | Sediments | 24.94 Dry | 25.37 Dry | 25.155 | 1.71 |
| | 6-1-4-1 | Sediments | 21.98 Dry | 23.16 Dry | 22.57 | 5.23 |
| | 7-1-3-1 | Sediments | 13.83 Dry | 14.59 Dry | 14.21 | 5.35 |
| Fe | 0-1-1-1 | Sediments | 24818 Dry | 30146 Dry | 27482 | 19.39 |
| | 3-2-1-1 | Sediments | 19730 Dry | 20744 Dry | 20237 | 5.01 |
| | 4-1-3-2 | Sediments | 10697 Dry | 11970 Dry | 11333.5 | 11.23 |
| | 5-1-3-1 | Sediments | 23905 Dry | 23523 Dry | 23714 | 1.61 |
| | 6-1-4-1 | Sediments | 9970 Dry | 12169 Dry | 11069.5 | 19.87 |
| | 7-1-3-1 | Sediments | 9922 Dry | 12304 Dry | 11113 | 21.43 |
| Hg | 0-1-1-1 | Sediments | < .1 Dry | < .1 Dry | 0.05 | 0 |
| | 3-2-1-1 | Sediments | < .1 Dry | < .1 Dry | 0.05 | 0 |
| | 4-1-3-2 | Sediments | < .1 Dry | < .1 Dry | 0.05 | 0 |
| | 5-1-3-1 | Sediments | < .1 Dry | < .1 Dry | 0.05 | 0 |
| | 6-1-4-1 | Sediments | < .1 Dry | < .1 Dry | 0.05 | 0 |
| | 7-1-3-1 | Sediments | < .1 Dry | < .1 Dry | 0.05 | 0 |
| Mn | 0-1-1-1 | Sediments | 689 Dry | 864 Dry | 776.5 | 22.54 |
| | 3-2-1-1 | Sediments | 979 Dry | 1149 Dry | 1064 | 15.98 |
| | 4-1-3-2 | Sediments | 492 Dry | 499 Dry | 495.5 | 1.41 |
| | 5-1-3-1 | Sediments | 924 Dry | 945 Dry | 934.5 | 2.25 |
| | 6-1-4-1 | Sediments | 339 Dry | 372 Dry | 355.5 | 9.28 |
| | 7-1-3-1 | Sediments | 438 Dry | 479 Dry | 458.5 | 8.94 |

DUPLICATES (Cont)

| Analyte | Sample Number | Sample Matrix | Initial Result | Duplicate Result | Relative % | |
|---------|---------------|---------------|----------------|------------------|------------|------------|
| | | | (ppm / %) | (ppm / %) | Average | Difference |
| Ni | 0-1-1-1 | Sediments | 20.9 Dry | 25.3 Dry | 23.1 | 19.05 |
| | 3-2-1-1 | Sediments | 20.6 Dry | 18.1 Dry | 19.35 | 12.92 |
| | 4-1-3-2 | Sediments | 11.3 Dry | 12 Dry | 11.65 | 6.01 |
| | 5-1-3-1 | Sediments | 29 Dry | 29 Dry | 29 | 0 |
| | 6-1-4-1 | Sediments | 13 Dry | 15.1 Dry | 14.05 | 14.95 |
| | 7-1-3-1 | Sediments | 17.5 Dry | 19.5 Dry | 18.5 | 10.81 |
| Pb | 0-1-1-1 | Sediments | 51.92 Dry | 45.33 Dry | 48.625 | 13.55 |
| | 3-2-1-1 | Sediments | 17.96 Dry | 16.72 Dry | 17.34 | 7.15 |
| | 4-1-3-2 | Sediments | 13.15 Dry | 11.82 Dry | 12.485 | 10.65 |
| | 5-1-3-1 | Sediments | 20.74 Dry | 21.95 Dry | 21.345 | 5.67 |
| | 6-1-4-1 | Sediments | 17.03 Dry | 17.77 Dry | 17.4 | 4.25 |
| | 7-1-3-1 | Sediments | 17.96 Dry | 19.54 Dry | 18.75 | 8.43 |
| Se | 0-1-1-1 | Sediments | < 1 Dry | < 1 Dry | 0.5 | 0 |
| | 3-2-1-1 | Sediments | < 1 Dry | < 1 Dry | 0.5 | 0 |
| | 4-1-3-2 | Sediments | < 1 Dry | < 1 Dry | 0.5 | 0 |
| | 5-1-3-1 | Sediments | < 1 Dry | < 1 Dry | 0.5 | 0 |
| | 6-1-4-1 | Sediments | < 1 Dry | < 1 Dry | 0.5 | 0 |
| | 7-1-3-1 | Sediments | < 1 Dry | < 1 Dry | 0.5 | 0 |
| Zn | 0-1-1-1 | Sediments | 88.6 Dry | 109.8 Dry | 99.2 | 21.37 |
| | 3-2-1-1 | Sediments | 76.2 Dry | 80.1 Dry | 78.15 | 4.99 |
| | 4-1-3-2 | Sediments | 56.4 Dry | 58.9 Dry | 57.65 | 4.34 |
| | 5-1-3-1 | Sediments | 103.9 Dry | 105.6 Dry | 104.75 | 1.62 |
| | 6-1-4-1 | Sediments | 55.3 Dry | 65.6 Dry | 60.45 | 17.04 |
| | 7-1-3-1 | Sediments | 50.7 Dry | 58.3 Dry | 54.5 | 13.94 |

REFERENCE MATERIALS

| Lab Sample | | | * Certified | 95% | | | |
|------------|--------|-------------|-------------|-----------------|----------|-----------|--------|
| Analyte | Number | S.R.M. ID | Reference | Confidence | Result | Percent | |
| Recovery | | | S.R.M. Name | Value (ppm / %) | Interval | (ppm / %) | |
| Al | BCSS-D | NRCC BCSS-1 | | | | 16219 Dry | |
| | BCSS-E | NRCC BCSS-1 | | | | 21388 Dry | |
| | BCSS-I | NRCC BCSS-1 | | | | 21829 Dry | |
| | BCSS-L | NRCC BCSS-1 | | | | 25020 Dry | |
| | BCSS-M | NRCC BCSS-1 | | | | 24391 Dry | |
| As | BCSS-D | NRCC BCSS-1 | Sediment | 11.1 Dry | 1.4 | 9.61 Dry | 86.58 |
| | BCSS-E | NRCC BCSS-1 | Sediment | 11.1 Dry | 1.4 | 9.58 Dry | 86.31 |
| | BCSS-I | NRCC BCSS-1 | Sediment | 11.1 Dry | 1.4 | 8.4 Dry | 75.68 |
| | BCSS-L | NRCC BCSS-1 | Sediment | 11.1 Dry | 1.4 | 8.19 Dry | 73.78 |
| | BCSS-M | NRCC BCSS-1 | Sediment | 11.1 Dry | 1.4 | 9.65 Dry | 86.94 |
| Cd | BCSS-D | NRCC BCSS-1 | Sediment | .25 Dry | .4 | .25 Dry | 100 |
| | BCSS-E | NRCC BCSS-1 | Sediment | .25 Dry | .4 | .25 Dry | 100 |
| | BCSS-I | NRCC BCSS-1 | Sediment | .25 Dry | .4 | .23 Dry | 92 |
| | BCSS-L | NRCC BCSS-1 | Sediment | .25 Dry | .4 | .24 Dry | 96 |
| | BCSS-M | NRCC BCSS-1 | Sediment | .25 Dry | .4 | .23 Dry | 92 |
| Cr | BCSS-D | NRCC BCSS-1 | Sediment | 123 Dry | 14 | 40.95 Dry | 33.29 |
| | BCSS-E | NRCC BCSS-1 | Sediment | 123 Dry | 14 | 51.13 Dry | 41.57 |
| | BCSS-I | NRCC BCSS-1 | Sediment | 123 Dry | 14 | 49.91 Dry | 40.58 |
| | BCSS-L | NRCC BCSS-1 | Sediment | 123 Dry | 14 | 53.12 Dry | 43.19 |
| | BCSS-M | NRCC BCSS-1 | Sediment | 123 Dry | 14 | 50.42 Dry | 40.99 |
| Cu | BCSS-D | NRCC BCSS-1 | Sediment | 18.5 Dry | 2.7 | 14.33 Dry | 77.46 |
| | BCSS-E | NRCC BCSS-1 | Sediment | 18.5 Dry | 2.7 | 18.52 Dry | 100.11 |
| | BCSS-I | NRCC BCSS-1 | Sediment | 18.5 Dry | 2.7 | 18.76 Dry | 101.41 |
| | BCSS-L | NRCC BCSS-1 | Sediment | 18.5 Dry | 2.7 | 18.57 Dry | 100.38 |
| | BCSS-M | NRCC BCSS-1 | Sediment | 18.5 Dry | 2.7 | 18.44 Dry | 99.68 |
| Fe | BCSS-D | NRCC BCSS-1 | | | | 23092 Dry | |

* Only certified analytes list a confidence interval - all others are considered reference values.

REFERENCE MATERIALS (Cont.)

| Lab Sample | | | * Certified 95% | | Result Interval | Percent (ppm / %) |
|------------|--------|-------------|-----------------|-----------------|--------------------|----------------------|
| Analyte | Number | S.R.M. ID | Reference | Confidence | | |
| Recovery | | | S.R.M. Name | Value (ppm / %) | | |
| <hr/> | | | | | | |
| Fe | BCSS-E | NRCC BCSS-1 | | | 21094 Dry | |
| | BCSS-I | NRCC BCSS-1 | | | 29562 Dry | |
| | BCSS-L | NRCC BCSS-1 | | | 30127 Dry | |
| | BCSS-M | NRCC BCSS-1 | | | 28568 Dry | |
| Hg | BEST-A | NRCC BEST-1 | Sediment | .092 Dry | .009 | < .1 Dry 108.7 |
| | BEST-D | NRCC BEST-1 | Sediment | .092 Dry | .009 | < .1 Dry 108.7 |
| | BEST-E | NRCC BEST-1 | Sediment | .092 Dry | .009 | < .1 Dry 108.7 |
| | BEST-I | NRCC BEST-1 | Sediment | .092 Dry | .009 | < .1 Dry 108.7 |
| | BEST-L | NRCC BEST-1 | Sediment | .092 Dry | .009 | < .1 Dry 108.7 |
| | BEST-M | NRCC BEST-1 | Sediment | .092 Dry | .009 | < .1 Dry 108.7 |
| Mn | BCSS-D | NRCC BCSS-1 | Sediment | 229 Dry | 15 | 180 Dry 78.6 |
| | BCSS-E | NRCC BCSS-1 | Sediment | 229 Dry | 15 | 228 Dry 99.56 |
| | BCSS-I | NRCC BCSS-1 | Sediment | 229 Dry | 15 | 231 Dry 100.87 |
| | BCSS-L | NRCC BCSS-1 | Sediment | 229 Dry | 15 | 229 Dry 100 |
| | BCSS-M | NRCC BCSS-1 | Sediment | 229 Dry | 15 | 219 Dry 95.63 |
| Ni | BCSS-D | NRCC BCSS-1 | Sediment | 55.3 Dry | 3.6 | 46.6 Dry 84.27 |
| | BCSS-E | NRCC BCSS-1 | Sediment | 55.3 Dry | 3.6 | 56.03 Dry 101.32 |
| | BCSS-I | NRCC BCSS-1 | Sediment | 55.3 Dry | 3.6 | 60.21 Dry 108.88 |
| | BCSS-L | NRCC BCSS-1 | Sediment | 55.3 Dry | 3.6 | 58.86 Dry 106.44 |
| | BCSS-M | NRCC BCSS-1 | Sediment | 55.3 Dry | 3.6 | 57.7 Dry 104.34 |
| Pb | BCSS-D | NRCC BCSS-1 | Sediment | 22.7 Dry | 3.4 | 21.94 Dry 96.65 |
| | BCSS-E | NRCC BCSS-1 | Sediment | 22.7 Dry | 3.4 | 21.99 Dry 96.87 |
| | BCSS-I | NRCC BCSS-1 | Sediment | 22.7 Dry | 3.4 | 20.55 Dry 90.53 |
| | BCSS-L | NRCC BCSS-1 | Sediment | 22.7 Dry | 3.4 | 21.35 Dry 94.05 |
| | BCSS-M | NRCC BCSS-1 | Sediment | 22.7 Dry | 3.4 | 21.05 Dry 92.73 |
| Se | BCSS-D | NRCC BCSS-1 | Sediment | .43 Dry | .06 | < 1 Dry 232.56 |
| | BCSS-E | NRCC BCSS-1 | Sediment | .43 Dry | .06 | < 1 Dry 232.56 |

* Only certified analytes list a confidence interval - all others are considered reference values.

REFERENCE MATERIALS (Cont.)

| Lab Sample | | S.R.M. ID | S.R.M. Name | * Certified | 95% | Result Interval | Percent (ppm / %) |
|------------|--------|-------------|-------------|-----------------|------------|--------------------|----------------------|
| Analyte | Number | | | Reference | Confidence | | |
| Recovery | | | | Value (ppm / %) | | | |
| Se | BCSS-I | NRCC BCSS-1 | Sediment | .43 Dry | .06 | < 1 Dry | 232.56 |
| | BCSS-L | NRCC BCSS-1 | Sediment | .43 Dry | .06 | < 1 Dry | 232.56 |
| | BCSS-M | NRCC BCSS-1 | Sediment | .43 Dry | .06 | < 1 Dry | 232.56 |
| Zn | BCSS-D | NRCC BCSS-1 | Sediment | 119 Dry | 12 | 102 Dry | 85.71 |
| | BCSS-E | NRCC BCSS-1 | Sediment | 119 Dry | 12 | 116.6 Dry | 97.98 |
| | BCSS-I | NRCC BCSS-1 | Sediment | 119 Dry | 12 | 120.5 Dry | 101.26 |
| | BCSS-L | NRCC BCSS-1 | Sediment | 119 Dry | 12 | 121.8 Dry | 102.35 |
| | BCSS-M | NRCC BCSS-1 | Sediment | 119 Dry | 12 | 115.3 Dry | 96.89 |

* Only certified analytes list a confidence interval - all others are considered reference values.

SPIKE RECOVERIES

| Analyte | Sample Number | Sample Matrix | Spike Level (ppm / %) | Amount Recovered (ppm / %) | * Spike / Background | Percent Recovery |
|---------|---------------|---------------|--------------------------|-------------------------------|-------------------------|---------------------|
| AVS | 0-1-1-2 | Sediments | 893.3 Dry | 693.8 Dry | 0.99 | 77.67 |
| | 3-2-1-2 | Sediments | 250.6 Dry | 233.4 Dry | 1.06 | 93.14 |
| | 4-2-1-1 | Sediments | 376.1 Dry | 345.8 Dry | 7.46 | 91.94 |
| | 5-1-3-2 | Sediments | 664.1 Dry | 611 Dry | 0.8 | 92 |
| | 6-1-4-2 | Sediments | 436.7 Dry | 407 Dry | 2.34 | 93.2 |
| | 7-1-3-2 | Sediments | 226.1 Dry | 206.4 Dry | 2.05 | 91.29 |
| As | 0-1-1-2 | Sediments | 7 Dry | 7.13 Dry | 1.07 | 101.86 |
| | 3-2-1-2 | Sediments | 7 Dry | 5.45 Dry | 1.05 | 77.86 |
| | 4-2-1-1 | Sediments | 6 Dry | 6.18 Dry | 0.73 | 103 |
| | 5-1-3-2 | Sediments | 7 Dry | 6.21 Dry | 1.29 | 88.71 |
| | 6-1-4-2 | Sediments | 7 Dry | 7.91 Dry | 2.3 | 113 |
| | 7-1-3-2 | Sediments | 7 Dry | 6.34 Dry | 0.97 | 90.57 |
| Cd | 0-1-1-2 | Sediments | 10 Dry | 9.93 Dry | 23.26 | 99.3 |
| | 3-2-1-2 | Sediments | 9 Dry | 11 Dry | 14.75 | 122.22 |
| | 4-2-1-1 | Sediments | 9 Dry | 10.43 Dry | 15.25 | 115.89 |
| | 5-1-3-2 | Sediments | 10 Dry | 9.94 Dry | 19.23 | 99.4 |
| | 6-1-4-2 | Sediments | 10 Dry | 10.24 Dry | 27.03 | 102.4 |
| | 7-1-3-2 | Sediments | 10 Dry | 10.65 Dry | 21.28 | 106.5 |
| Cr | 0-1-1-2 | Sediments | 53 Dry | 61.13 Dry | 3.15 | 115.34 |
| | 3-2-1-2 | Sediments | 52 Dry | 50.21 Dry | 3.3 | 96.56 |
| | 4-2-1-1 | Sediments | 51 Dry | 41.61 Dry | 3.58 | 81.59 |
| | 5-1-3-2 | Sediments | 53 Dry | 53.23 Dry | 2.01 | 100.43 |
| | 6-1-4-2 | Sediments | 55 Dry | 72.53 Dry | 6.71 | 131.87 |
| | 7-1-3-2 | Sediments | 55 Dry | 65.33 Dry | 2.73 | 118.78 |
| Cu | 0-1-1-2 | Sediments | 56 Dry | 53.48 Dry | 1.84 | 95.5 |
| | 3-2-1-2 | Sediments | 54 Dry | 52.08 Dry | 2.5 | 96.44 |
| | 4-2-1-1 | Sediments | 38 Dry | 41.65 Dry | 2.36 | 109.61 |
| | 5-1-3-2 | Sediments | 55 Dry | 49.5 Dry | 1.86 | 90 |

* For a spike to be a valid measure of method accuracy, this ratio must be higher than 1.0.

SPIKE RECOVERIES (Cont.)

| Analyte | Sample Number | Sample Matrix | Spike Level (ppm / %) | Amount Recovered (ppm / %) | * Spike / Background | Percent Recovery |
|---------|---------------|---------------|--------------------------|-------------------------------|-------------------------|---------------------|
| Cu | 6-1-4-2 | Sediments | 57 Dry | 59.07 Dry | 2.5 | 103.63 |
| | 7-1-3-2 | Sediments | 57 Dry | 59.78 Dry | 2.6 | 104.88 |
| Hg | 0-1-1-2 | Sediments | 1.27 Dry | 1.28 Dry | 12.7 | 101.57 |
| | 3-2-1-2 | Sediments | 1.27 Dry | 1.36 Dry | 12.7 | 107.09 |
| | 4-2-1-1 | Sediments | 1.27 Dry | 1.36 Dry | 12.7 | 107.09 |
| | 5-1-3-2 | Sediments | 1.27 Dry | 1.32 Dry | 12.7 | 103.94 |
| | 6-1-4-2 | Sediments | 1.27 Dry | 1.33 Dry | 12.7 | 104.72 |
| | 7-1-3-2 | Sediments | 1.27 Dry | 1.28 Dry | 12.7 | 100.79 |
| Mn | 0-1-1-2 | Sediments | 813 Dry | 865 Dry | 0.94 | 106.4 |
| | 3-2-1-2 | Sediments | 659 Dry | 870 Dry | 0.58 | 132.02 |
| | 4-2-1-1 | Sediments | 370 Dry | 440 Dry | 0.69 | 118.92 |
| | 5-1-3-2 | Sediments | 671 Dry | 743 Dry | 0.59 | 110.73 |
| | 6-1-4-2 | Sediments | 697 Dry | 755 Dry | 2 | 108.32 |
| | 7-1-3-2 | Sediments | 694 Dry | 803 Dry | 1.11 | 115.71 |
| Ni | 0-1-1-2 | Sediments | 167 Dry | 209 Dry | 7.91 | 125.15 |
| | 3-2-1-2 | Sediments | 162 Dry | 172.6 Dry | 10.06 | 106.54 |
| | 4-2-1-1 | Sediments | 160 Dry | 138.4 Dry | 9.58 | 86.5 |
| | 5-1-3-2 | Sediments | 165 Dry | 169.3 Dry | 5.34 | 102.61 |
| | 6-1-4-2 | Sediments | 172 Dry | 184.4 Dry | 18.11 | 107.21 |
| | 7-1-3-2 | Sediments | 171 Dry | 184.9 Dry | 6.5 | 108.13 |
| Pb | 0-1-1-2 | Sediments | 248 Dry | 394.72 Dry | 9.87 | 159.16 |
| | 3-2-1-2 | Sediments | 241 Dry | 245.24 Dry | 12.49 | 101.76 |
| | 4-2-1-1 | Sediments | 237 Dry | 255.9 Dry | 12.67 | 107.97 |
| | 5-1-3-2 | Sediments | 245 Dry | 233.98 Dry | 10.63 | 95.5 |
| | 6-1-4-2 | Sediments | 255 Dry | 272.56 Dry | 14.03 | 106.89 |
| | 7-1-3-2 | Sediments | 254 Dry | 253.74 Dry | 10.51 | 99.9 |
| Se | 0-1-1-2 | Sediments | 46 Dry | 51.78 Dry | 46 | 112.57 |
| | 3-2-1-2 | Sediments | 45 Dry | 42.51 Dry | 45 | 94.47 |

* For a spike to be a valid measure of method accuracy, this ratio must be higher than 1.0

SPIKE RECOVERIES (Cont.)

| Analyte | Sample Number | Sample Matrix | Spike Level (ppm / %) | Amount Recovered (ppm / %) | * Spike / Background | Percent Recovery |
|-----------|---------------|---------------|--------------------------|-------------------------------|-------------------------|---------------------|
| Se | 4-2-1-1 | Sediments | 44 Dry | 46.56 Dry | 44 | 105.82 |
| | 5-1-3-2 | Sediments | 46 Dry | 43.21 Dry | 46 | 93.93 |
| | 6-1-4-2 | Sediments | 47 Dry | 46.61 Dry | 47 | 99.17 |
| | 7-1-3-2 | Sediments | 47 Dry | 55.87 Dry | 47 | 118.87 |
| Zn | 0-1-1-2 | Sediments | 283 Dry | 377.5 Dry | 2.97 | 133.39 |
| | 3-2-1-2 | Sediments | 273 Dry | 251.9 Dry | 3.7 | 92.27 |
| | 4-2-1-1 | Sediments | 268 Dry | 218.1 Dry | 4.08 | 81.38 |
| | 5-1-3-2 | Sediments | 278 Dry | 260.2 Dry | 2.2 | 93.6 |
| | 6-1-4-2 | Sediments | 289 Dry | 308.8 Dry | 5.19 | 106.85 |
| | 7-1-3-2 | Sediments | 288 Dry | 311.5 Dry | 3.63 | 108.16 |

* For a spike to be a valid measure of method accuracy, this ratio must be higher than 1.0.

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Purchase Order: 85830-2-3836

COMMENTS (RESULT MODIFIERS AND QA/QC COMMENTS)

| Analyte | Sample Number | Result Modifier |
|---------|------------------|-----------------|
| ----- | ----- | ----- |

NO DATA EXIST FOR THIS SECTION.

QA/QC Comments

APPROVAL,EC

ANALYTICAL METHODS

Method
Code

Method Description

002 LABORATORY: Geochemical & Environmental Research Group, Texas A&M

Tissue, Sediment and Water Mercury

- II. Mercury was determined by EPA method 245.5 with minor revisions. Sediment samples can be analyzed either freeze dried or on a wet basis. Sediment samples are homogenized by mixing before subsampling. The tissue samples were homogenized in the original sample containers with a Tekar Tisumizer and subsampled. Water samples are acidified (0.5% v/v with high purity nitric acid, HNO₃) in the original sample bottle. For sediments a 0.5 to 1.0 gram sample (dry weight) was used. For tissues a 1.5 to 2.0 gram sample (wet weight) was used. For water the sample size is 20 ml.

For tissue and sediment, the sample is weighed into a 50 ml polypropylene centrifuge tube. 2.5 ml of concentrated sulfuric acid (H₂SO₄) and 1.5 ml of concentrated nitric acid (HNO₃) were added and the samples heated in a water bath at 90 C for 15 min. After cooling 10 ml of distilled water and 15 ml of mixture of 3.3% (w/w) potassium permanganate (KMnO₄), and 1.7% (w/w) potassium persulfate (K₂S₂O₈) were added to each tube and the samples heated in a water bath at 90 C for 30 min. After cooling 5 ml of 10% (w/w) hydroxylamine hydrochloride (NH₂OH HC1) was added to reduce excess permanganate and the volume brought to 35 ml with distilled water.

For water samples, the sample is weighed into a 50 ml polypropylene centrifuge tube, 1 ml of concentrated H₂SO₄ is added and the solution mixed vigorously with a vortex stirrer. Then 4.5 ml of the KMnO₄/K₂S₂O₈ is added and the resulting mixture heated in a 90 C water bath for 2 hours. After cooling, 1.5 ml of a 10% (w/w) hydroxylamine hydrochloride (NH₂OH HC1) solution is added, sample volume adjusted to a constant volume with distilled water and the resulting solution mixed vigorously.

ANALYTICAL METHODS (Cont.)

Method
Code

Method Description

002

Mercury is determined by a modification of the method of Hatch and Ott (1968). A portion of the digest solution is placed in a sealed container. To this is added 0.4 ml of 10% (w/w) stannous chloride (SnCl₂). Mercury is reduced to the elemental state and aerated from solution into an atomic absorption spectrophotometer where its concentration is measured.

References

1. "USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis." Document Number ILMO1.0 USEPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.
2. "Interim Method for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," USEPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, Aug. 1977, Revised Oct. 1980.
3. Hatch, W.R. and Ott, W.L., "Determination of Sub-Microgram Quantities of Mercury in Solution by a Flameless Atomic Absorption Technique", Analytical Chemistry 40,14 p 2085-2087 (1968).

003 **LABORATORY:** Geochemical & Environmental Research Group, Texas A&M

% Dry Weight

- III. Approximately 1 gram of wet sample is weighed into a clean, labeled, preweighed 10 ml beaker. The beaker is placed in a forced air oven at approximately 75 degrees Celsius for 24 hours. The beaker with the dry sample is then weighed and the % dry weight is calculated by the formula:

$$\frac{(\text{wt. dry sample and beaker}) - (\text{wt. beaker})}{(\text{wt. dry sample and beaker})} (100)$$

ANALYTICAL METHODS (Cont.)

**Method
Code**

Method Description

003

(wt. wet sample and beaker) - (wt. beaker)

007 LABORATORY: Geochemical & Environmental Research Group, Texas A&M

Trace Metal-Sediment

VII. Sediments are digested with aqua regia (3:1 HCl:HNO₃) in glass beakers on a hotplate and diluted to volume with distilled water. Metals in the digestate are determined by 3 techniques, depending upon concentration and element. Mercury is determined by cold vapor atomic absorption spectrometry (AAS), in which Sn²⁺ is used to reduce HgO. Arsenic, selenium, cadmium, and lead are determined by graphite furnace AAS, in which electrical heating is used to produce an atomic cloud. Remaining elements (and Cd or Pb when in high concentration) are determined by atomic emission using an argon plasma.

013 LABORATORY: Geochemical & Environmental Research Group, Texas A&M

Acid Volume Sulfide (AVS)

XIII. Acid volatile sulfide (AVS) is defined as sulfides that are converted to H₂S upon exposure to 1N HCl at room temperature for 1 hour. Approximately 10 grams of wet sediment are acidified with 1N HCl, and reactive sulfides that are converted to gaseous H₂S are trapped as Ag₂S and measured gravimetrically.